WEATHERING BEHAVIOUR OF BAGASSE FIBER REINFORCED POLYMER COMPOSITE

A THESIS SUBMITTED IN PARTIAL FULFILMENT OF THE REQUIREMENTS FOR THE DEGREE OF

Master of Technology (Research)

In

Mechanical Engineering

By

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National Institute of Technology

Rourkela - 769008

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CERTIFICATE

This is to certify that thesis entitled, "**WEATHERING BEHAVIOUR OF BAGASSE FIBER REINFORCED POLYMER COMPOSITE"** submitted by **Mr SURAJ KUMAR MEHAR** in partial fulfillment of the requirements for the award of Master of Technology Degree in Mechanical Engineering at National Institute of Technology, Rourkela (Deemed University) is an authentic work carried out by him under our supervision and guidance.

To the best of our knowledge, the matter embodied in this thesis has not been submitted to any other university/ institute for award of any Degree or Diploma.

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CONTENTS

ABSTRACT

In recent years the natural fiber composites have attracted substantial importance as a potential structural material. The attractive features of natural fibers like jute, sisal, coir and banana have been their low cost, light weights, high specific modulus, renewability and biodegradability. Natural fibers are lignocellulosic in nature. These composites are gaining importance due to their non-carcinogenic and bio-degradable nature. The natural fiber composites can be very cost effective material especially for building and construction industry. However in many instances residues from traditional crops such as rice husk or sugarcane bagasse or from the usual processing operations of timber industries do not meet the requisites of being long fibers. Bagasse contains about 40% cellulose, 30% hemicellulose, and 15% lignin. The present use of bagasse is mainly as a fuel in the sugar cane mill furnaces. It is felt that the value of this agricultural residue can be upgraded by bonding with resin to produce composites suitable for building materials.

Keeping this in view the present work has been undertaken to develop a polymer matrix composite (epoxy resin) using bagasse fiber as reinforcement and to study its mechanical properties and environmental performance. The composites were prepared with different volume fraction of bagasse fibers. Experiments have been conducted under laboratory conditions to asses the effect of different environment such as subzero, steam, saline water and natural conditions on the mechanical properties of the composites. Flexural strength of the composites was evaluated by three point bend test as per ASTM D2344-84. The volume fraction of composites having greater mechanical properties was taken for the second phase of experimentation. The second phase of experiment involves treatment of bagasse fiber with alkali and acetone. To asses the change in mechanical properties due to the treated fiber the composite was again subjected to different environment such as subzero, steam, saline water and natural conditions. Micro structural examinations were also made to get an idea about the effect of treated and untreated fibers on the mechanical properties of the composites.

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INTRODUCTION

CHAPTER – 1

1.1 BACKGROUND

India endowed with an abundant availability of natural fiber such as Jute, Coir, Sisal, Pineapple, Ramie, Bamboo, Banana etc. has focused on the development of natural fiber composites primarily to explore value-added application avenues. Such natural fiber composites are well suited as wood substitutes in the housing and construction sector. The development of natural fiber composites in India is based on two pronged strategy of preventing depletion of forest resources as well as ensuring good economic returns for the cultivation of natural fibers.

The developments in composite material after meeting the challenges of aerospace sector have cascaded down for catering to domestic and industrial applications. Composites, the wonder material with light-weight; high strength-to-weight ratio and stiffness properties have come a long way in replacing the conventional materials like metals, wood etc. The material scientists all over the world focused their attention on natural composites reinforced with Jute, Sisal, Coir, Pineapple etc. primarily to cut down the cost of raw materials.

1.2 WHY A COMPOSITE?

Over the last thirty years composite materials, plastics and ceramics have been the dominant emerging materials. The volume and number of applications of composite materials have grown steadily, penetrating and conquering new markets relentlessly. Modern composite materials constitute a significant proportion of the engineered materials market ranging from everyday products to sophisticated niche applications.

While composites have already proven their worth as weight-saving materials, the current challenge is to make them cost effective. The efforts to produce economically attractive composite components have resulted in several innovative manufacturing techniques currently being used in the composites industry. It is obvious, especially for composites, that the improvement in manufacturing technology alone is not enough to overcome the cost hurdle. It is essential that there be an integrated effort in design, material,

process, tooling, quality assurance, manufacturing, and even program management for composites to become competitive with metals.

The composites industry has begun to recognize that the commercial applications of composites promise to offer much larger business opportunities than the aerospace sector due to the sheer size of transportation industry. Thus the shift of composite applications from aircraft to other commercial uses has become prominent in recent years.

Increasingly enabled by the introduction of newer polymer resin matrix materials and high performance reinforcement fibres of glass, carbon and aramid, the penetration of these advanced materials has witnessed a steady expansion in uses and volume. The increased volume has resulted in an expected reduction in costs. High performance FRP can now be found in such diverse applications as composite armoring designed to resist explosive impacts, fuel cylinders for natural gas vehicles, windmill blades, industrial drive shafts, support beams of highway bridges and even paper making rollers. For certain applications, the use of composites rather than metals has in fact resulted in savings of both cost and weight. Some examples are cascades for engines, curved fairing and fillets, replacements for welded metallic parts, cylinders, tubes, ducts, blade containment bands etc.

Further, the need of composite for lighter construction materials and more seismic resistant structures has placed high emphasis on the use of new and advanced materials that not only decreases dead weight but also absorbs the shock & vibration through tailored microstructures. Composites are now extensively being used for rehabilitation/ strengthening of pre-existing structures that have to be retrofitted to make them seismic resistant, or to repair damage caused by seismic activity.

Unlike conventional materials (e.g., steel), the properties of the composite material can be designed considering the structural aspects. The design of a structural component using composites involves both material and structural design. Composite properties (e.g. stiffness, thermal expansion etc.) can be varied continuously over a broad range of values under the control of the designer. Careful selection of reinforcement type enables finished product characteristics to be tailored to almost any specific engineering requirement.

Whilst the use of composites will be a clear choice in many instances, material selection in others will depend on factors such as working lifetime requirements, number of items to be produced (run length), complexity of product shape, possible savings in assembly

costs and on the experience & skills the designer in tapping the optimum potential of composites. In some instances, best results may be achieved through the use of composites in conjunction with traditional materials.

1.3 DEFINITION OF COMPOSITE

The most widely used meaning is the following one, which has been stated by Jartiz [1] "Composites are multifunctional material systems that provide characteristics not obtainable from any discrete material. They are cohesive structures made by physically combining two or more compatible materials, different in composition and characteristics and sometimes in form".

The weakness of this definition resided in the fact that it allows one to classify among the composites any mixture of materials without indicating either its specificity or the laws which should given it which distinguishes it from other very banal, meaningless mixtures.

Kelly [2] very clearly stresses that the composites should not be regarded simple as a combination of two materials. In the broader significance; the combination has its own distinctive properties. In terms of strength to resistance to heat or some other desirable quality, it is better than either of the components alone or radically different from either of them.

Beghezan [3] defines as "The composites are compound materials which differ from alloys by the fact that the individual components retain their characteristics but are so incorporated into the composite as to take advantage only of their attributes and not of their short comings", in order to obtain improved materials.

Van Suchetclan [4] explains composite materials as heterogeneous materials consisting of two or more solid phases, which are in intimate contact with each other on a microscopic scale. They can be also considered as homogeneous materials on a microscopic scale in the sense that any portion of it will have the same physical property.

1.4 CHARACTERISTICS OF THE COMPOSITES

Composites consist of one or more discontinuous phases embedded in a continuous phase. The discontinuous phase is usually harder and stronger than the continuous

phase and is called the ‘reinforcement‘ or ‘reinforcing material’, whereas the continuous phase is termed as the ‘ matrix’.

Properties of composites are strongly dependent on the properties of their constituent materials, their distribution and the interaction among them. The composite properties may be the volume fraction sum of the properties of the constituents or the constituents may interact in a synergistic way resulting in improved or better properties. Apart from the nature of the constituent materials, the geometry of the reinforcement (shape, size and size distribution) influences the properties of the composite to a great extent. The concentration distribution and orientation of the reinforcement also affect the properties.

The shape of the discontinuous phase (which may by spherical, cylindrical, or rectangular cross-sanctioned prisms or platelets), the size and size distribution (which controls the texture of the material) and volume fraction determine the interfacial area, which plays an important role in determining the extent of the interaction between the reinforcement and the matrix.

Concentration, usually measured as volume or weight fraction, determines the contribution of a single constituent to the overall properties of the composites. It is not only the single most important parameter influencing the properties of the composites, but also an easily controllable manufacturing variable used to alter its properties.

1.5 CLASSIFICATION

Composite materials can be classified in different ways [5]. Classification based on the geometry of a representative unit of reinforcement is convenient since it is the geometry of the reinforcement which is responsible for the mechanical properties and high performance of the composites. A typical classification is presented in table1.1. The two broad classes of composites are (1) Particulate composites and (2) Fibrous composites.

1.5.1 Particulate Composites

As the name itself indicates, the reinforcement is of particle nature (platelets are also included in this class). It may be spherical, cubic, tetragonal, a platelet, or of other regular or irregular shape, but it is approximately equiaxed. In general, particles are not very effective in improving fracture resistance but they enhance the stiffness of the composite to a

limited extent. Particle fillers are widely used to improve the properties of matrix materials such as to modify the thermal and electrical conductivities, improve performance at elevated temperatures, reduce friction, increase wear and abrasion resistance, improve machinability, increase surface hardness and reduce shrinkage.

1.5.2 Fibrous composites

A fiber is characterized by its length being much greater compared to its cross-sectional dimensions. The dimensions of the reinforcement determine its capability of contributing its properties to the composite. Fibers are very effective in improving the fracture resistance of the matrix since a reinforcement having a long dimension discourages the growth of incipient cracks normal to the reinforcement that might other wise lead to failure, particularly with brittle matrices.

Man-made filaments or fibers of non polymeric materials exhibit much higher strength along their length since large flaws, which may be present in the bulk material, are minimized because of the small cross-sectional dimensions of the fiber. In the case of polymeric materials, orientation of the molecular structure is responsible for high strength and stiffness.

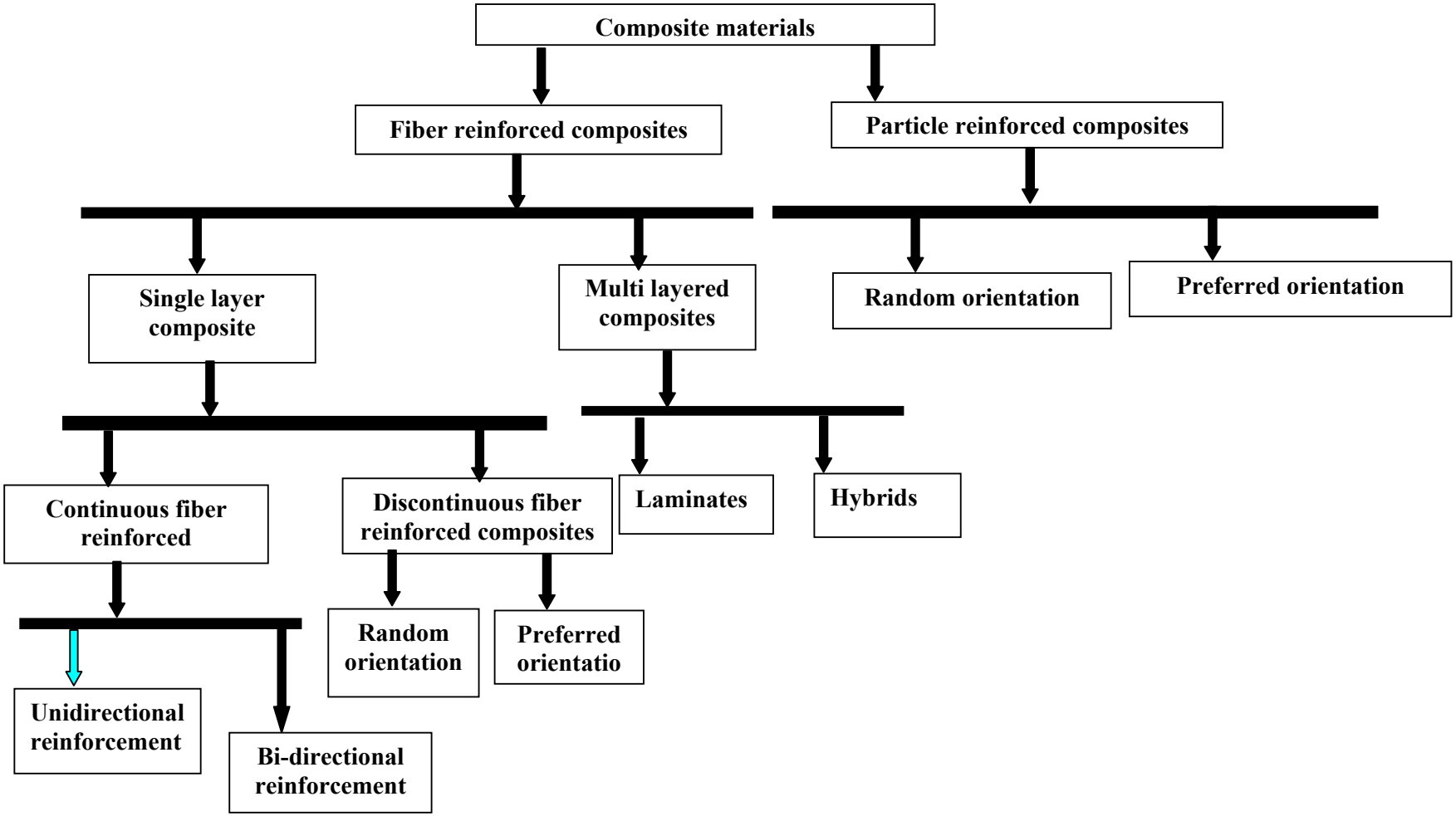


Fig 1 .1 Classification of composites

Fibers, because of their small cross- sectional dimensions, are not directly usable in engineering applications. They are, therefore, embedded in matrix materials to form fibrous composites. The matrix serves to bind the fibers together, transfer loads to the fibers, and protect them against environmental attack and damage due to handling. In discontinuous fiber reinforced composites, the load transfer function of the matrix is more critical than in continuous fiber composites.

1.6 COMPONENTS OF A COMPOSITE MATERIAL

In its most basic form a composite material is one, which is composed of at least two elements working together to produce material properties that are different to the properties of those elements on their own. In practice, most composites consist of a bulk material (the 'matrix'), and a reinforcement of some kind, added primarily to increase the strength and stiffness of the matrix.

1.6.1 Role of matrix in a composite

Many materials when they are in a fibrous form exhibit very good strength property but to achieve these properties the fibres should be bonded by a suitable matrix. The matrix isolates the fibres from one another in order to prevent abrasion and formation of new surface flaws and acts as a bridge to hold the fibres in place. A good matrix should possess ability to deform easily under applied load, transfer the load onto the fibres and evenly distributive stress concentration.

1.6.2 Materials used as matrices in composites

In its most basic form a composite material is one, which is composed of at least two elements working together to produce material properties that are different to the properties of those elements on their own. In practice, most composites consist of a bulk material (the matrix) and a reinforcement of some kind, added primarily to increase the strength and stiffness of the matrix.

(a) BULK PHASES

(1) Metal Matrices

Metal matrix composites possess some attractive properties, when compared with organic matrices. These include (i) strength retention at higher temperatures, (ii) higher

transverse strength, (iii) better electrical conductivity, (iv) superior thermal conductivity, (v) higher erosion resistance etc. However, the major disadvantage of metal matrix composites is their higher densities and consequently lower specific mechanical properties compared to polymer matrix composites. Another notable difficulty is the high-energy requirement for fabrication of such composites.

(2) Polymer Matrices

A very large number of polymeric materials, both thermosetting and thermoplastic, are used as matrix materials for the composites. Some of the major advantages and limitations of resin matrices are shown in Table 1.1.

Generally speaking, the resinous binders (polymer matrices) are selected on the basis of adhesive strength, fatigue resistance, heat resistance, chemical and moisture resistance etc. The resin must have mechanical strength commensurate with that of the reinforcement. It must be easy to use in the fabrication process selected and also stand up to the service conditions. Apart from these properties, the resin matrix must be capable of wetting and penetrating into the bundles of fibres which provide the reinforcement, replacing the dead air spaces therein and offering those physical characteristics capable of enhancing the performance of fibres.

(3) Ceramic Matrices

Ceramic fibres, such as alumina and SiC (Silicon Carbide) are advantageous in very high temperature applications, and also where environment attack is an issue. Since ceramics have poor properties in tension and shear, most applications as reinforcement are in the particulate form (e.g. zinc and calcium phosphate). Ceramic Matrix Composites (CMCs) used in very high temperature environments, these materials use a ceramic as the matrix and reinforce it with short fibres, or whiskers such as those made from silicon carbide and boron nitride.

(b) REINFORCEMENT

The role of the reinforcement in a composite material is fundamentally one of increasing the mechanical properties of the neat resin system. All of the different fibres used in composites have different properties and so affect the properties of the composite in different ways. For most of the applications, the fibres need to be arranged into some form of

sheet, known as a fabric, to make handling possible. Different ways for assembling fibers into sheets and the variety of fiber orientations possible to achieve different characteristics.

(c) INTERFACE

It has characteristics that are not depicted by any of the component in isolation. The interface is a bounding surface or zone where a discontinuity occurs, whether physical, mechanical, chemical etc. The matrix material must "wet" the fiber. Coupling agents are frequently used to improve wet ability. Well "wetted" fibres increase the interface surfaces area. To obtain desirable properties in a composite, the applied load should be effectively transferred from the matrix to the fibres via the interface. This means that the interface must be large and exhibit strong adhesion between fibres and matrix. Failure at the interface (called debonding) may or may not be desirable.

1.7 TYPES OF COMPOSITE MATERIALS

The composite materials are broadly classified into the following categories as shown in fig 1.2 (a-e).

1.7.1 Fiber-Reinforced Composites

Reinforced-composites are popularly being used in many industrial applications because of their inherent high specific strength and stiffness. Due to their excellent structural performance, the composites are gaining potential also in tribological applications. In this type composite the second phase is in the form of fibers dispersed in the matrix which could be either plastic or metal. The volume fraction (Vf) varies from a few percentage to as high as 70%. Usually the fiber reinforcement is done to obtain high strength and high modulus. Hence it is necessary for the fibers to posses higher modulus than the matrix material, so that the load is transferred to the fiber from the matrix more effectively.

1.7.2 Dispersion Hardened Material

In this type of material, fine particles of sizes ranging from 0.01μm to 0.14μm are dispersed in matrix. Their concentration varies from 1% to 15% by volume. These fine particles impede dislocation movement in the material and therefore result in very high strength. Also these materials posses improved high temperature strength and creep resistance.

1.7.3 Particulate composite

In this type of composites, 1μm to 200μm size particles are dispersed in the matrix and volume fraction is generally between 0.01 Vf to 0.85 Vf.

1.8 NATURAL FIBER COMPOSITES: Initiative in Product Development

Natural fibres are lignocellulosic in nature. These composites are gaining importance due to their non-carcinogenic and bio-degradable nature [6-9]. The natural fiber composites can be very cost effective material especially for building and construction industry (panels, false ceilings, partition boards etc.) packaging, automobile and railway coach interiors and storage devices. This also can be a potential candidate in making of composites, especially for partial replacement of high cost glass fibers for low load bearing applications. However in many instances residues from traditional crops such as rice husk or sugarcane bagasse or from the usual processing operations of timber industries do not meet the requisites of being long fibers. This biomass left over are abundant, and their use as a particulate reinforcement in resin matrix composite is strongly considered as a future possibility.

Large varieties of sugar cane grow abundantly in many parts of India. Cane is crushed in a series of mills (Fig 1.3), each consisting of at least three heavy rollers. Due to the crushing, the cane stalk will break in small pieces, and subsequent milling will squeeze the juice out. The juice is collected and processed for production of sugar. The resulting crushed and squeezed cane stalk, named bagasse, is considered to be a by-product of the milling process [10]. Bagasse is essentially a waste product that causes mills to incur additional disposal costs.

Bagasse is a fibrous residue that remains after crushing the stalks, and contains short fibers (Fig. 1.4). It consists of water, fibers, and small amounts of soluble solids. Percent contribution of each of these components varies according to the variety, maturity, method of harvesting, and the efficiency of the crushing plant. Table 1.2 shows a typical bagasse composition [10].

Bagasse is mainly used as a burning raw material in the sugar cane mill furnaces. The low caloric power of bagasse makes this a low efficiency process. Also, the sugar cane mill

management encounters problems regarding regulations of “clean air” from the Environmental Protection Agency, due to the quality of the smoke released in the atmosphere. Presently 85% of bagasse production is burnt. Even so, there is an excess of bagasse. Usually this excess is deposited on empty fields altering the landscape. Approximately 9% of bagasse is used in alcohol (ethanol) production. Ethanol is not just a good replacement for the fossil fuels, but it is also an environmentally friendly fuel. Apart from this, ethanol is a very versatile chemical raw material from which a variety of chemicals can be produced [11]. But again, due to the low level of sucrose left in bagasse, the efficiency of the ethanol production is quite low.

With increasing emphasis on fuel efficiency, natural fibers such as bagasse based composites enjoying wider applications in automobiles and railway coaches & buses for public transport system. There exist an excellent opportunity in fabricating bagasse based composites towards a wide array of applications in building and construction such boards and blocks as reconstituted wood, flooring tiles etc. Value added novel applications of natural fibers and bagasse based composites would not go in a long way in improving the quality of life of people engaged in bagasse cultivation, but would also ensure international market for cheaper substitution.

Natural fibers have the advantages of low density, low cost, and biodegradability. However, the main disadvantages of natural fibers and matrix and the relative high moisture sorption. Therefore, chemical treatments are considered in modifying the fiber surface properties.

A better understanding of the chemical composition and surface adhesive bonding of natural fiber is necessary for developing natural fiber is necessary for developing natural fiber-reinforced composites. The components of natural fibers include cellulose, hemicellulose, lignin, pectin, waxes and water soluble substances. The composition of selected natural fibers is shown in Table 1.3 [12, 13].The composition may differ with the growing condition and test methods even for the same kind of fiber. Cellulose is a semicrystalline polysaccharide made up of D-glucopyranose units linked together by β-(1-4)-glucosidic bonds [13]. And the large amount of hydroxyl group in cellulose gives natural fiber hydrophilic properties when used to reinforce hydrophobic matrices; the result is a very poor interface and poor resistance to moisture absorption [14]. Hemicellulose is strongly bound to cellulose fibrils presumably by hydrogen bonds. Hemicellulosic polymers are

branched, fully amorphous and have a significantly lower molecular weight than cellulose. Because of its open structure containing many hydroixyl and acetyl groups, hemicellulose is partly soluble in water and hygroscopic [15]. Lignins are amorphous, highly complex, mainly aromatic, polymers of phenylpropane units [13] but have the least water sorption of the natural fiber components [15].

Because of the low interfacial properties between fiber and polymer matrix often reduce their potential as reinforcing agents due to the hydrophilic nature of natural fibers, chemical modifications are considered to optimize the interface of fibers. Chemicals may activate hydroxyl groups or introduce new moieties that can effectively interlock with the matrix. The development of a definitive theory for the mechanism of bonding by chemicals in composites is a complex problem. Generally, chemical coupling agents are molecules possessing two functions. The first function is to react with hydroxyl group of cellulose and the second is to react with functional groups of the matrix.

Visualizing the increased rate of utilization of natural fibers the present work has been undertaken to develop a polymer matrix composite (epoxy resin) using bagasse fiber as reinforcement and to study its mechanical properties and environmental performance. The composites are to be prepared with different volume fraction of bagasse fibers. Then they will be subjected to different environmental treatment such as, subzero, steam, saline water and natural conditions for various time lengths. The change in weight, volume and dimensions are to be studied for various treatments. Shear strength of the composites has to be evaluated by three point bend test. The volume fraction of composites having greater mechanical properties is to be taken for the second phase of experimentation. The second group of samples will involve bagasse fiber surface treatments, namely (i) unwashed bagasse, (ii) unwashed and treated with acetone and alkali (iii) washed and treated with acetone and alkali. The treated fibers along with the samples with highest mechanical properties with washed fibers already manufactured will be taken for comparison of mechanical properties subjecting them to different environmental treatments. Micro structural examinations (SEM) will be made to ascertain the fracture behavior of the composite.

Keeping all this in view the entire work has been divided into five chapters.

In the second chapter work related to present investigations available in literatures are presented.

The third and fourth chapters represent the preparation of specimens for the composites, their treatments and characterization.

In fifth chapter conclusions have been drawn from the above studies mentioning the scope for future work.

Table 1.1

Advantages and limitations of polymeric matrix materials

Advantages	Limitations
Low densities	Low transverse strength
Good corrosion resistance	Low operational temperature limits
Low thermal conductivities	
Low electrical conductivities	
Translucence	
Aesthetic Color effects	

Table 1.2

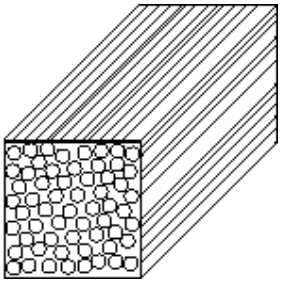
Average Bagasse Composition

ITEM	%
Moisture	49.0
Soluble Solids	2.3
Fiber	48.7
Cellulose	41.8
Hemicelluloses	28
Lignin	21.8

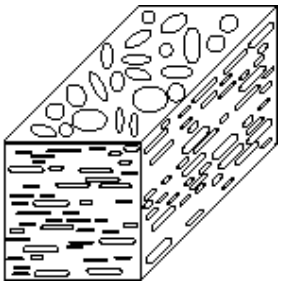
Table -1.3

Chemical composition of selected common natural fibers

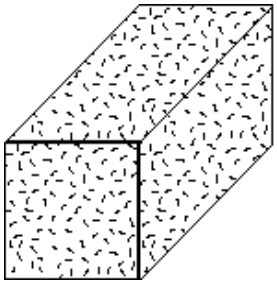
Types of fiber	Cellulose (%)	Lignin (%)	Hemicellulose (%)	Pectin (%)	Ash (%)
A. Bast fiber					
Fiber flax	71	22	18.6-20.6	2.3	-
Seed flax	43-47	21-23	24-26	-	5
Kenaf	31-57	15-19	21.5-23	-	2-5
Jute	45-71.5	12-26	13.6-21	0.2	0.5-2
Hemp	57-77	3.7-13	14-22.4	0.9	0.8
Remie	68.6-91	0.6-0.7	5-16.7	1.9	-
B.Core fiber					
Kenaf	37-49	15-21	18-24	-	2-4
Jute	41-48	21-24	18-22	-	0.8
C.Leaf fiber					
Abaca	56-63	7-9	15-17	-	3
Sisal	47-78	7-11	10-24	10	0.6-1
Henequen	77.6	13.1	4-8	-	-



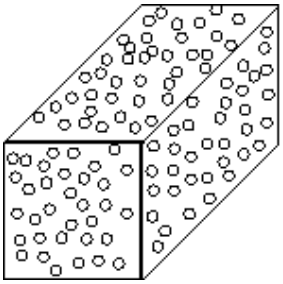
(a) Random fiber (short fiber) reinforced composites



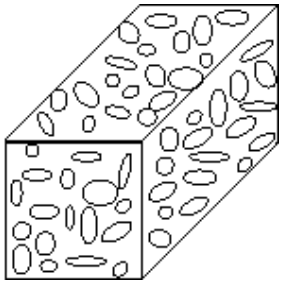
(b) Particles as the reinforcement (Particulate composites)



(c) Continuous fiber (long fiber) reinforced Composites



(d) Flat flakes as the reinforcement (Flake composites)



(e) Fillers as the reinforcement (Filler composites)

Fig 1.2 (a-e) Classification of composite materials

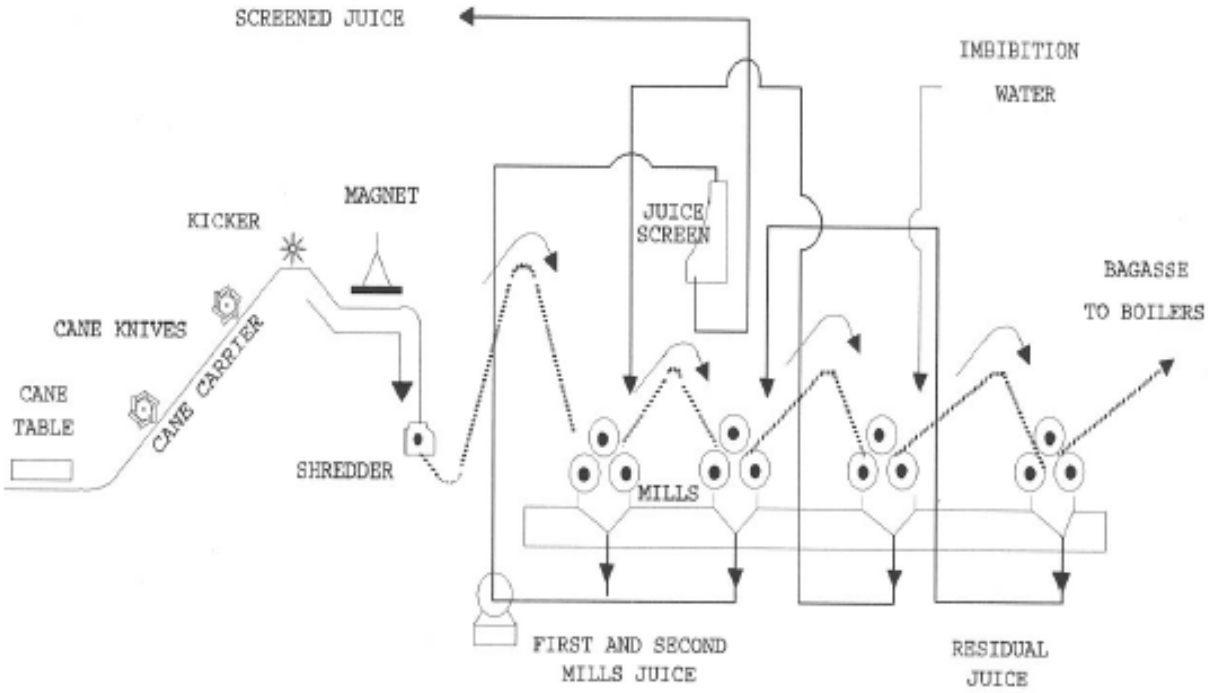


Fig. 1.3 Current technological process for extraction of sugar juice from cane in a sugar cane mill

Fig. 1.4 Bagasses

Chapter 2

LITERATURE SURVEY

CHAPTER – 2

2. 1 INTRODUCTION

The literature survey is carried out as a part of the thesis work to have an overview of the production processes, properties and weathering behavior of polymer matrix composites. Composite structures have shown universally a savings of at least 20% over metal counterparts and a lower operational and maintenance cost [16]. As the data on the service life of composite structures is becoming available, it can be safely said that they are durable, maintain dimensional integrity, resist fatigue loading and are easily maintainable and repairable. Composites will continue to find new applications, but the large scale growth in the market place for these materials will require less costly processing methods and the prospect of recycling [17] will have to be solved [18].

Composites materials have emerged as a major class of structural elements and are either used or being considered as substitutions for metals/traditional material in aerospace, automotive and other industries. The outstanding features of fiber reinforced polymer composites (FRPs) are their high specific stiffness, high specific strength and controlled anisotropy, which make them very attractive structural materials. Other advantages of composites are light weight, good corrosion resistance, impact resistance, fatigue strength and flexibility in design capabilities. A unique feature of composites is that the characteristics of the finished product can be tailored to a specific engineering requirement by a careful selection of matrix and reinforcement type. FRP composite materials consist of two or more chemically distinct constituents have a distinct interface separating them. It has a unique combination of properties that are noticeably different from the constituent properties. Generally, a discontinuous phase (reinforcement) is embedded into a continuous phase (matrix). Polymer based composite materials (PMC) or FRP constitutes a major category of composites materials with a wide range of applications. They offer very attractive properties, which can be tailored to the specific requirements by careful selection the fiber, matrix, fiber configuration (short, long, strength, woven, braided, laminated, etc.) and fiber surface treatment. PMCs exhibit desirable physical and chemical properties that include lightweight coupled with high stiffness and strength along the direction of the reinforcing fiber,

dimensional stability, temperature and chemical resistance and relatively easy processing. The role of matrix in a fiber-reinforced composite is to

(a) Transfer stresses between the fibers
(b) Provide a barrier against an adverse environment
(c) Protect the surface of fibers from mechanical abrasion.

2.2 MATERIAL SELECTION

2.2.1 Matrix Material

Because it is much more than dispersing glue in PMC, the matrix alloy should be chosen only after giving careful consideration to its chemical compatibility with the reinforcement, to its ability to wet the reinforcement, and to its own characteristics properties and processing behavior [17, 19].

2.2.2 Why Polymer Matrix Selection?

Polymers are structurally much more complex than metals or ceramic. They are cheap and can be easily processed. On the other hand, polymers have lower strength and modulus and lower temperature use limits. Prolonged exposure to ultraviolet light and some solvents can cause the degradation of polymer properties. Because of predominantly covalent bonding, polymers are generally poor conductors of heat and electricity. Polymers, however, are generally more resistant to chemicals than are metals. Structurally, polymers are giant chainlike molecules (hence the name macromolecules) with covalently bonded carbon atoms forming the backbone of the chain. The process of forming large molecules from small ones is called polymerization; that is, polymerization is the process of joining many monomers, the basic building blocks, together to form polymer. Polymers used to manufacture advanced PMCs are of two basic types thermoset and thermoplastics resins.

a) Thermoset resins

Thermoset resins dominate the advanced composites industry today, while thermoplastics have only a minor role. It requires addition of a curing agent or hardner and impregnation onto a reinforcing material, followed by a curing step to produce a cured or finished part. Some of the more common thermoset resins are described briefly here.

Epoxy resins are relatively low molecular weight monomers with low shrinkage during cure. They can be partially cured and stored in that state. The cured epoxy resins have high chemical and corrosion resistance, good mechanical thermal properties. However, they are more expensive compared to polyester resin. The second of the essential ingredients of an advanced composite system is the curing agent or hardner. These compounds are very important because they control the reaction rate and determine the performance characteristics of the finished part. Since these compounds act as catalysts for the reaction, they must contain active sites on their molecules. Some of the most commonly used curing agent in the advanced composite industry is the aromatic amines like 4.4 methylene-dianiline (MDA) and 4.4-sulfonylianiline (DDS)

Unsaturated polyester resins are most widely used due to their good mechanical properties, corrosion resistance, low weight and low cost. These consist of linear polymer chains dissolved in styrene monomer. These polymer chains have reactive sites resulting from the incorporation of the anhydrous forms of unsaturated dicarboxylic acids (e.g. maleic anhydride). The reactive unsaturation sites on the polymer chains react and crosslinking with the styrene monomer via a free radical reaction. This reaction is usually initiated by the addition of a peroxide catalyst, such a methyl ethyl ketone peroxide (MEKP). The presence of an accelerator in the resin, such as cobalt octate, speed the reaction at a give temperature . The addition of heat significantly speeds up this cross-linking reaction.

Polyurethanes are another group of resin used in advanced composite process. These compounds are formed by reaction the polyol component with an isocynate compound, typically toluene diisocynate (TDI). Methylene diisocynate (MDI) and hexamethylene diisocynate (HDI) are also widely used. These are used to manufacture fiber reinforced structural foams. Phenolic and amino resins are used extensively in aircraft interiors because of the exceptional low smoke and heat release properties in the event of a fire.

b) Thermoplastic resins

Thermoplastics resins require only heat and pressure to form the finished part. Unlike the thermoset resins, the thermoplastics resins can usually be reheated and reformed into another shape, if desired. Common examples of thermoplastics resins are polyethylene, polystyrene, nylon, polycarbonate, polysulfone, polyphenylene sulfine etc. Polyamides,

Polyimide, PEEK are relatively newcomers to the composite industry and are used for high temperature applications. These resins have better thermal stability and flame resistance than the epoxy resins. Polyamide based composites have excellent retention of strength in how-wet environment but they are brittle and have a very low elongation at break.

2.2.3 Reinforcement

Reinforcement increases the strength, stiffness and the temperature resistance capacity and lowers the density of PMC. In order to achieve these properties the selection depends on the type of reinforcement, its method of production and chemical compatibility with the matrix and the following aspects must be considered while selecting the reinforcement material.

- Size – diameter and aspect ratio
- Shape – Chopped fiber, whisker, spherical or irregular particulate, flake, etc:
- Surface morphology – smooth or corrugated and rough:
- Poly – or single crystal
- Structural defects – voids, occluded material, second phase
- Surface chemistry
- Impurities
- Inherent properties – strength, modulus and density.

2.2.4 Reinforcement Materials

Fibers as reinforcing material offer two advantages. Firstly, the bulk material is always stronger when produced as small diameter fibers due to the natural exclusion of large scale defects. Secondly, the fiber configuration allows the tailoring of properties in specific directions. Fibers are added to the resin system to provide strength to the finished part. The selection of reinforcing material is based on the properties desired in the finished product. Fibers used in advanced composite manufacture come in various forms, such as yarns, roving, chopped strands, woven fabric and mats. Each of these has its own special application. In processes such as filament winding or pultrusion, yarns and roving are used. When performs are used in parts manufacture, woven fabric or mats are required.

2.3 FABRICATION METHODS OF PMCs

There are two general divisions of composites manufacturing processes: **open molding** and **closed molding.** With open molding, the gel coat and laminate are exposed to the atmosphere during the fabrication process. In closed molding, the composite is processed in a two-part mold set, or within a vacuum bag. There are a variety of processing methods within the open and closed molding categories:

a) Open Molding Method: Hand Lay-Up, Spray-Up, Filament Winding

b) Closed Molding Method: Compression molding, Pultrusion, Vacuum Bag Molding, Vacuum Infusion Processing, Resin Transfer Molding (RTM)

2.3.1 Open Molding

Open molding process is saturating fiber reinforcement with resin, using manual rollout techniques to consolidate the laminate and removing the entrapped air. A major factor in this operation is the transfer of resin from a drum or storage tanks to the mold. The means used to transport the resin, in many cases, characterizes the specific process method.

a) Hand Lay –Up

Hand lay-up is an open molding method suitable for making a wide variety of composites products including: boats, tanks bathware, housings, truck/auto components, architectural products and many other products ranging from very small to very large. Production volume per mold is low; however, it is feasible to produce substantial production quantities using multiple molds. Simple, single-cavity molds of fiberglass composites construction are generally used.

Molds can range from very small to very large and are low cost in the spectrum of soft composites molds.

Gel coat is first applied to the mold using a spray gun for a high-quality surface. When the gel coat has cured sufficiently, roll stock fiberglass reinforcement is manually

placed on the mold. The lamination resin is applied by pouring, brushing, spraying, or using a paint roller. FRP rollers, paint rollers, or squeegees are used to consolidate the laminate, thoroughly wetting the reinforcement, and removing entrapped air. Subsequent layers of fiberglass reinforcement are added to build laminate thickness (Fig 2.1).

Simplest method offering low-cost tooling, simple processing and wide range of part sizes are the major advantages of this process. Design changes are readily made. There is a minimum investment in equipment. With skilled operators, good production rates consistent quality is obtainable.

b) Spray Lay-Up

Spray-up or chopping is similar to hand lay-up in its suitability for making boats, tanks, transportation components and tub/shower units in a large variety of shapes and sizes. A chopped laminate has good conformability and is sometimes faster than hand lay-up in molding complex shapes. In the spray-up process the operator controls thickness and consistency, therefore the process is more operator dependent than hand lay-up. Although production volume per mold is low, it is feasible to produce substantial production quantities using multiple molds. As with hand lay-up, gel coat is first applied to the mold prior to spray-up of the substrate laminate.

Continuous strand glass roving and catalyzed resin are fed through a chopper gun, which deposits the resin-saturated “chop” on the mold as shown in fig 2.2. The laminate is then rolled to thoroughly saturate the glass strands and compact the chop. Additional layers of chop laminate are added as required for thickness.

c) Filament Winding

Filament winding is an automated open molding process that uses a rotating mandrel as the mold. The male mold configuration produces a finished inner surface and a laminated rough surface on the outside diameter of the product. Filament winding results in a high degree of fiber loading, which provides high tensile strengths in the manufacture of hollow, generally cylindrical products such as chemical and fuel storage tanks, pipes, stacks, pressure vessels, and rocket motor cases. Mandrels of suitable size and shape, made of steel or

aluminium form the inner surface of the hollow part. Some mandrels are collapsible to facilitate part removal.

Figure 2.3 shows the schematic picture of a typical filament winding process. Continuous strand roving is fed through a resin bath and would onto a rotating mandrel. The roving feed runs on a trolley that traverses the length of the mandrel. The filament is laid down in a predetermined geometric pattern to provide maximum strength in the directions required. When sufficient layers have been applied, the laminate is cured on the mandrel. The molded part is then stripped from the mandrel. Equipment is available for filament winding on a continuous basis with two axes winding for pressure cylinders. This process makes high strength-to-weight ratio laminates and provides a high degree of control over uniformity and fiber orientation. The filament winding process can be used to make structures, which are highly engineered and meet strict tolerances. Because filament winding is automated, the labor factor for filament winding is lower than other open molding processes.

2.3.2 Closed Molding Method

a) Compression Molding

Compression molding is a high-volume, high-pressure method suitable for molding complex, fiberglass-reinforced plastic parts on a rapid cycle time. There are several types of compression molding including: sheet molding compound (SMC) which are, bulk molding compound (BMC), thick molding compound (TMC), and wet lay-up compression molding.

Compression molding tooling consists of heated metal molds mounted in large presses. Tooling is usually machined steel or cast alloy molds that can be in either single or multiple-cavity configurations. Steel molds are hardened and sometimes chrome plated for enhanced durability. The molds are heated using steam, hot oil, or electricity. Side cores, provisions for inserts, and other refinements are often employed. Mold materials include cast of forged steel, cast iron, and cast aluminum.

The mold set is mounted in a hydraulic or mechanical molding press. The molds are heated to 2500 to 4000 F. A weight charge of molding compound is placed in the open mold as shown in fig 2.4. The two halves of the mold are closed and pressure is applied. Depending

on thickness, size, and shape of the part, curing cycles range from less than a minute to about five minutes. The mold is opened and the finished part is removed. Typical parts include: automobile components, appliance housings and structural components, furniture, electrical components, and business machine housings and parts.

Compression molding produces fast molding cycles and high part uniformity. The process can be automated. Good part design flexibility and features such as inserts, ribs, bosses, and attachments can be molded in .Good surface finishes are obtainable, contributing to lower part finishing cost. Subsequent trimming and machining operations are minimized in compression molding.

b) Pultrusion

Pultrusion is a continuous process for the manufacture of products having a constant cross section, such as rod stock, structural shapes, beams channels, pipe, tubing, fishing rods, and golf club shafts. Pultrusion produces profiles with extremely high fiber loading, thus pultruded products have high structural properties. Hardened steel dies are machined and include a perform area to do the initial shaping of the resin- saturated roving. The dies include heating which can be electric or hot oil. The latest pultrusion technology uses direct injection dies, in which the resin is introduced inside the die, rather than through an external resin bath, which may be called as partial RTM.

Continuous strand fiberglass roving, mat, cloth, or surfacing veil is impregnated in a resin bath, then pulled (pul-trusion) through a steel die, by a powerful tractor mechanism (Refer fig 2.5). The steel die consolidates the saturated reinforcement, sets the shape of the stock, and controls the fiber/resin ratio. The die is heated to rapidly cure the resin. Many creels (balls) of roving are positioned on a rack, and a complex series of tensioning devices and roving guides direct the roving into the die.

The process is a continuous operation that can be readily automated. It is adaptable to both simple and complex cross-sectional shapes. Very high strengths are possible due to the fiber loading and labor costs are low.

c) Vacuum Bag Molding

The mechanical properties of open-mold laminates can be improved with vacuum bagging. By reducing the pressure inside the vacuum bag, external atmospheric pressure exerts force on the bag. The pressure on the laminate removes entrapped air, excess resin, and compacts the laminate. Vacuum bagging can be used with wet-lay laminates and prepreg advanced composites. In wet lay-up bagging the reinforcement is saturated using hand lay-up, then the vacuum bag is mounted on the mold and used to compact the laminate and remove air voids. In the case of pre-impreg advanced composites molding, the prepreg material is laid-up on the mold, the vacuum bag is mounted and the mold is heated or the mold is placed in an autoclave that applies both heat and external pressure, adding to the force of atmospheric pressure. The prepreg-vacuum bag-autoclave method is most often used to create advanced composites used in aircraft and military products. Molds are similar to those used for conventional open-mold processes.

In the simplest form of vacuum bagging, a flexible film (PVA, nylon, mylar, or polyethylene) is placed over the wet lay-up, the edges sealed, and a vacuum drawn. A more advanced form of vacuum bagging places a release film over the laminate, followed by a bleeder ply of fiberglass cloth, non-woven nylon, polyester cloth, or other material that absorbs excess resin from the laminate. Fig 2.6 shows the schematic picture of vacuum bag molding process. A breather ply of a non-woven fabric is placed over the bleeder ply, and the vacuum bag is mounted over the entire assembly. Pulling a vacuum from within the bag uses atmospheric pressure to eliminate voids and force excess resin from the laminate. The addition of pressure further results in high fiber concentration and provides better adhesion between layers of sandwich construction. When laying non-contoured sheets of PVC foam or balsa into a female mold, vacuum bagging is the technique of choice to ensure proper secondary bonding of the core to the outer laminate.

Vacuum bag processing can produce laminates with a uniform degree of consolidation, while at the same time removing entrapped air, thus reducing the finished void content. Structures fabricated with traditional hand lay-up techniques can become resin rich and vacuum bagging can eliminate the problem. Additionally, complete fiber wet-out can be accomplished if the process is done correctly. Improved core-bonding is also possible with vacuum bag processing.

d) Vacuum Infusion Processing

Vacuum infusion is a variation of vacuum bagging where the resin is introduced into the mold after the vacuum has pulled the bag down and compacted the laminate. The method is defined as having lower than atmospheric pressure in the mold cavity. The reinforcement and core material are laid-up dry in the mold. This is done by hand and provides the opportunity to precisely position the reinforcement. When the resin is pulled into the mold the laminate is already compacted; therefore, there is no room for excess resin. Very high resin to glass ratio are possible with vacuum infusion and the mechanical properties of the laminate are superior. Vacuum infusion is suitable to mold very large structures and is considered a low volume molding process. Molds are similar to those used for conventional open-mold processes.

The mold may be gel coated in the tradition fashion. After the gel coat cures, the dry reinforcement is positioned in the mold. This includes all the plies of the laminate and core material if required. A perforated release film is placed over the dry reinforcement. Next a flow media consisting of a course mesh or a “crinkle” ply is positioned, and perforated tubing is positioned as a manifold to distribute resin across the laminate. The vacuum bag is then positioned and sealed at the mold perimeter. A tube is connected between the vacuum, bag and the resin container. A vacuum is plied to consolidate the laminate and the resin is pulled into the mold (fig 2.7).

Vacuum infusion can produce laminates with a uniform degree of consolidation, producing high strength, lightweight structures. This process uses the same low cost tooling as open molding and requires minimal equipment. Very large structures can be fabricated using this method. Vacuum infusion offers a substantial emissions reduction compared to either open molding or wet lay-up vacuum bagging.

e) Resin Transfer Molding

Resin transfer molding is an intermediate volume molding process for producing composites. The RTM process is to inject resin under pressure into a mold cavity. Vacuum assist can be used to enhance resin flow in the mold cavity. RTM can use a wide variety of tooling, ranging from low cost composite molds to temperature controlled metal tooling.

RTM can utilize either “hard” or “soft” tooling, depending upon the expected duration of the run. Soft tooling would be either polyester or epoxy molds, while hard tooling may consist of cast machined aluminum, electroformed nickel shell, or machined steel molds. RTM can take advantage of the broadest range of tooling.

Figure 2.8 shows the picture of resin transfer molding process of polyester resin with peroxide catalyst. The mold set is gel coated conventionally, if required. The reinforcement (and core material) is positioned in the mold and the mold is closed and clamped. The resin is injected under pressure, using mix/meter injection equipment, and the part is cured in the mold. The reinforcement can be either a preform or pattern cut roll stock material. Performs are reinforcement that is pre-formed in a separate process and can be quickly positioned in the mold. RTM can be done at room temperature; however, heated molds are required to achieve fast cycle times and product consistency.

This closed molding process produces parts with two finished surfaces. By laying up reinforcement material dry inside the mold, any combination of materials and orientation can be used, including 3-D reinforcements. Part thickness is determined by the tool cavity.

Fiber reinforced composites are popularly being used in many industrial applications because of their high specific strength and stiffness. Due to their excellent structural performance, these composites are gaining potential also in tribological applications [20]. In this type of composites the second phase is in the form of fibers dispersed in the matrix which could be either plastic or metal. Usually the fiber reinforcement is done to obtain high strength and high modulus. Hence it is necessary for the fibers to possess higher modulus than the matrix material, so the load is transferred to the fiber from the matrix more effectively. Natural fibers to the maximum extent fulfill these criteria and therefore have drawn world wide attention as a potential reinforcement material for the composites.

Natural fibers currently used as reinforcements in composite materials include jute, sisal, pineapple, abaca and coir [21-30]. The abundance and low cost of natural fibers combined with their low density and reduced wear on processing machinery makes these fibers suitable for use in composite materials. Synthetic fibers such as carbon or glass fibers have constant diameters; smooth surfaces and considerable rigidity. On the other hand, natural fibers can be flexible, have variable diameters along the length of each fiber and have

rough surfaces. Natural fibers are also sensitive to temperature and moisture and usually have irregular cross section.

The main chemical constituents of bagasse are hemi cellulose and lignin. Hemi cellulose and cellulose are present in the form of holocellulose in bagasse, which contributes more than 70 % of the total chemical constituent present in bagasse. Another important chemical constituent present in bagasse is lignin. Lignin acts as a binder for the cellulose fibers and also behaves as an energy storage system.

Usamani etal [31] describes the evolution of five water soluble phenolic resin as binders at 5 percent concentration, for oriented and random reinforced bagasse composite. They tried to determine the amount of resin retained during processing when these phenolics were precipitated on to bagasse fibers.

Monteiro SN. Rodriquez etal. [32] tries to use the sugar cane bagasse waste as reinforcement to polymeric resins for fabrication of low cost composites. They reported that composites with homogeneous microstructures could be fabricated and mechanical properties similar to wooden agglomerates can be achieved.

A.Vazquez, V.A.Dominguez etal. [33] in their work reported the processing and properties of bagasse fiber-polypropylene composites. Four different chemical treatments were done on fiber to improve interface adhesion with the thermoplastic matrix namely isocyanate, acrylic acid, mercerization and washing with alkaline solution. They observed that tensile strength and elongation at break of polypropylene matrix composite decrease with incorporation of bagasse fiber without treatment. Their result shows that the best results were obtained on materials with treated fibers.

Hassan etal. [34, 35] have converted the bagasse into a thermo formable material through esterification of the fiber matrix. Their results shows that on reacting bagasse with succinic anhydride (SA) in the absence of solvent, ester content up to about 48% could be obtained. The dimensional stability and mechanical properties of the composites prepared from the esterified fibers were reported in this work. They found dimensional stability to be dependent on the total ester and monoester/diester content of esterified fibers and increased with increasing total ester and monoester content of the fibers and the mechanical properties

(bending strength, tensile strength, and hardness) were enhanced with increasing monoester contents.

Paiva etal. [36] analyzed the impact strength and hardness of sugarcane bagasse-resol composites and showed that impact strength increased and hardness diminished as the fiber volume fraction increased.

Jane M. F.Paiva, E.Frollini [37] used short sugar cane fibers as reinforcement to obtain fiber reinforced composites. Lignin extracted from sugarcane bagasse was used as a partial substitute of phenol (40w/w) in resole phenolic matrices. They characterized the composite by mechanical tests such as impact, DMTA and hardness tests. The results as a whole showed that it is feasible to replace part of phenol by lignin in phenolic matrices without loss of properties.

K.Bilba, M.A.Arsene, A. Ouensanga [38] have studied the feasibility of bagasse fiber/cement composites. The influence of different parameters on the setting of the composite material has been studied. This study shows a retarding effect of lignin on the setting of the composite, for small amount of heat treated bagasse (200^0C) the behaviour of the composite is closely the same as the classical cement or cellulose/cement composite. They reported that the effect of mixing raw whole bagasse to commercial cement delays the setting times and decreases the maximum hydration temperature of setting.

M.V.de sousa etal. [39] studied the effect of three processing parameters on the flexural mechanical behaviour of chopped bagasse poly-ester composite. The parameters evaluated were: the size of the chopped material, the pretreatment derived from the previous processing of the bagasse material on mills and the molding pressure. The results obtained by them enable the selection of the best combination of bagasse origin, size and molding pressure.

Shinichi Shibata, Yong Cao, and Isao Fukumoto [40] in their work investigated experimentally the flexural modulus of the press molding composites made from bagasse fiber and biodegradable resin. They have also numerically predicted flexural modulus by using Cox's model. They conclude that up to 65% volume fraction of reinforcement flexural modulus increases. Decrease in the flexural modulus was found below 3 mm at the fiber length in the experimental and same trend was shown in the numerical prediction.

Mechanical properties of biodegradable composites reinforced with bagasse fiber with alkali treatment have been reported by Cao etal. [41]. Approximately 13 % improvement in tensile strength, 14 % in flexural strength and 30% in impact strength has been reported.

Yu-Tao zheng etal. [42] in their work focused on the effect of benzoic acid as the surface modifier on the mechanical properties of the bagasse fiber. They found in their experiment that the ratio of PVC/BF, the content of benzoic acid, and processing temperature had a significant effect on the mechanical properties of the composite, which was examined by the orthogonal optimal method. Their result also indicates that the interface modifier improved significantly on the tensile strength and little on the impact strength of the composite.

Recently Tayeb [43, 44] studied the potential use of bagasse fiber for tribological applications. His results shows that bagasse fiber composite can be a promising composite in friction and wear environment which can be competitive to glass fiber reinforced polyester composite.

After reviewing the existing literature available on natural fiber composites, particularly bagasse fiber composites efforts are put to understand the basic needs of the growing composite industry. The conclusions drawn from this is that, the success of combining vegetable natural fibers with polymer matrices results in the improvement of mechanical properties of the composites compared with the matrix materials. These fillers are cheap and nontoxic, can be obtained from renewable sources, and are easily recyclable. Moreover, despite their low strength, they can lead to composites with high specific strengths because of their low density.

Thus the priority of this work is to prepare Polymer Matrix Composites (PMCs) using bagasse fiber (waste from sugarcane industry) as reinforcement material. To improve the interfacial strength between the fiber and the matrix, the surface modification of the bagasse fiber has to be done by chemical treatment. The composite will then be subjected to different weathering treatments like steam, saline and subzero conditions. The flexural strength of the composite will be evaluated using three point bend test.

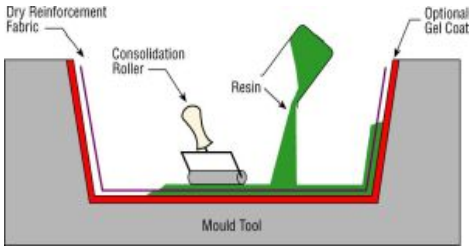


Fig 2.1 Hand Lay-Up Technique

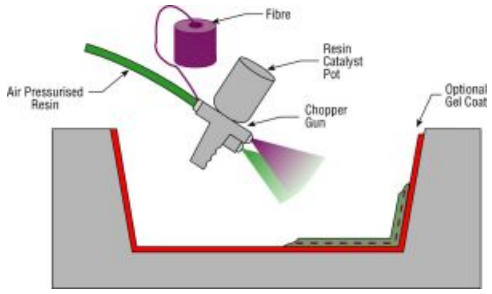


Fig 2.2 Spray up Technique

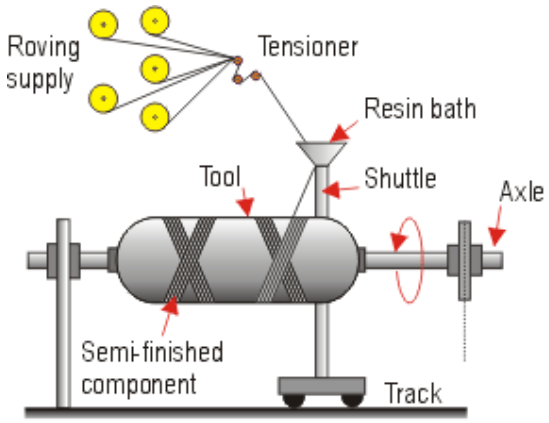


Fig 2.3 Filament Winding Process

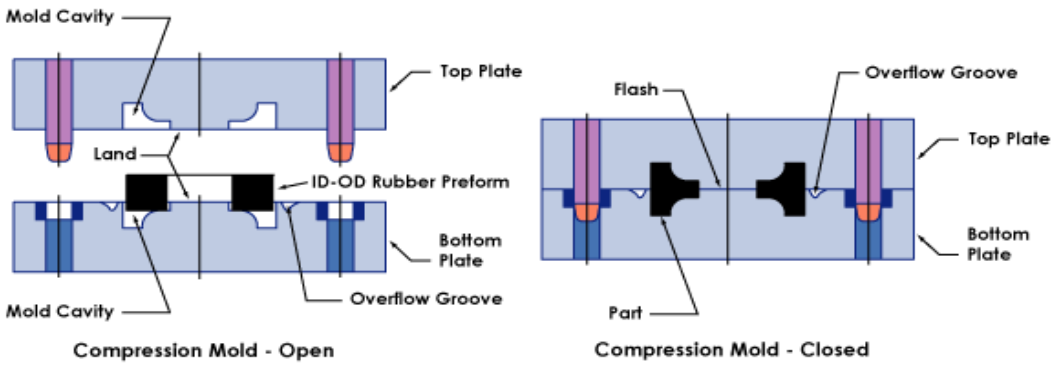


Fig.2.4 Compression Molding Technique

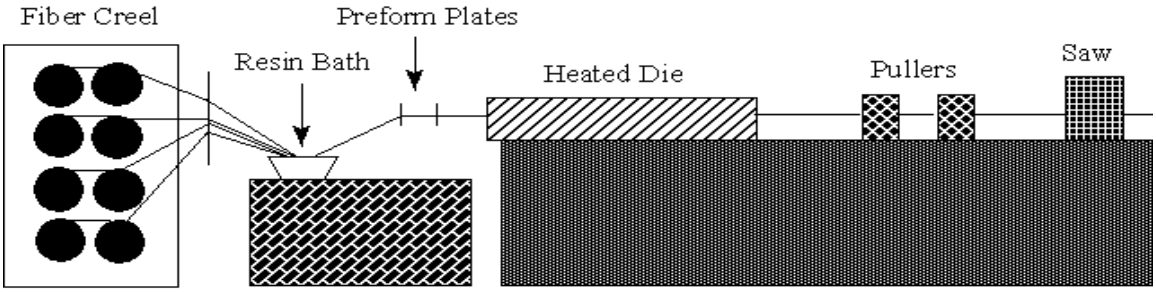


Fig 2.5 Pultrusion Process

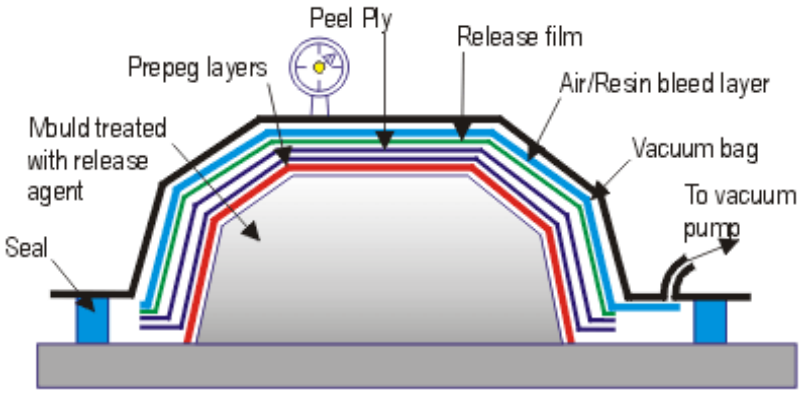


Fig 2.6 Vacuum Bag Molding

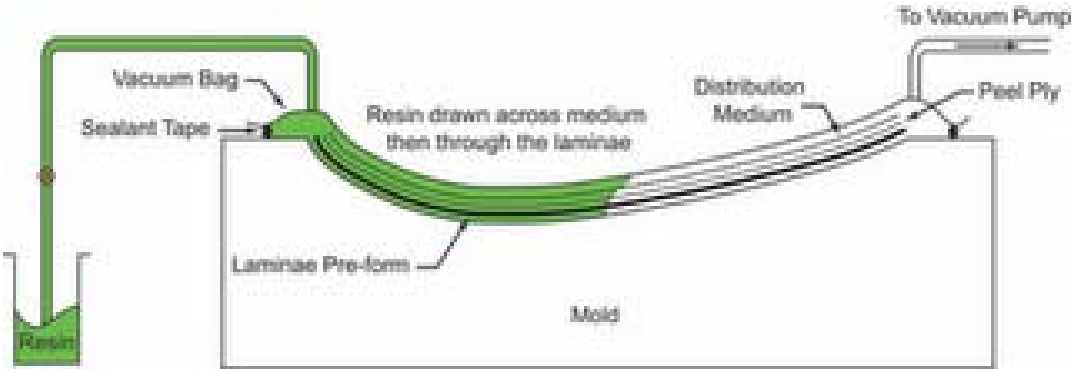


Fig 2.7 Vacuum Infusion Process

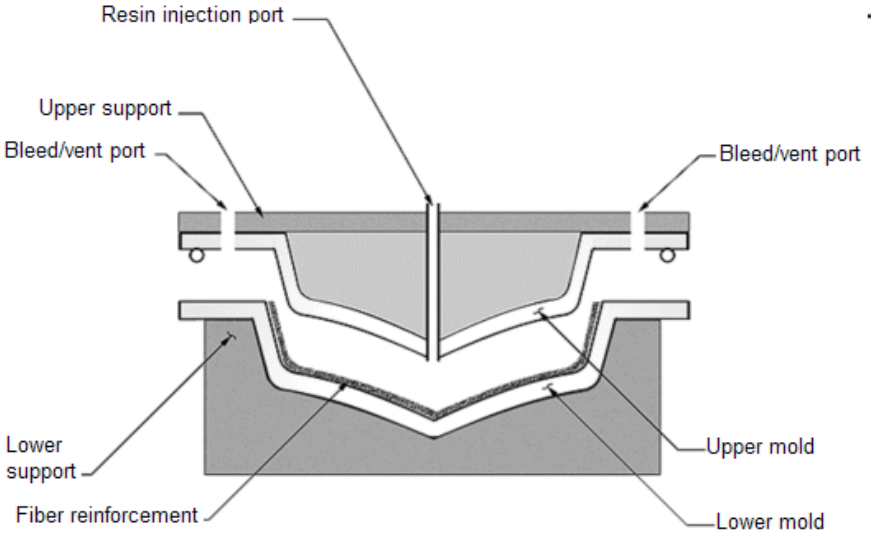


Fig 2.8 Resin Transfer Molding

Chapter 3

EFFECT OF ENVIRONMENT ON MECHANICAL PROPERTIES OF BAGASSE FIBER COMPOSITE

CHAPTER – 3

3. MATERIALS AND METHODS

3.1 RAW MATERIALS

Raw materials used in this experimental work are listed below:

1. Natural fiber (Bagasse)
2. Epoxy resin
3. Hardener

3.1.1 Bagasse fiber

The sugar cane bagasse is a residue widely generated in high proportions in the agro-industry. It is a fibrous residue of cane stalks left over after the crushing and extraction of juice from the sugar cane. Bagasse is generally gray-yellow to pale green in colour. It is bulky and quite non uniform in particle size. The sugar cane residue bagasse is an under utilized, renewable agricultural material that consist of two distinct cellular constituents. The first is a thick walled, relatively long, fibrous fraction derived from the rind and fibro-vascular bundles dispersed through out the interior of the stalk. The second is a pith fraction derived from the thin walled cells of the ground tissue.

The main chemical constituents of bagasse are cellulose, hemicellulose and lignin. Hemicellulose and cellulose are present in the form of hollow cellulose in bagasse which contributes to about 70 % of the total chemical constituents present in bagasse. Another important chemical constituent present in bagasse is lignin. Lignin acts as a binder for the cellulose fibers and also behaves as an energy storage system. Fig.3.1 is the SEM micrograph of the cross section of a raw bagasse fiber, which exhibits the cellular structure of the fiber.

In the present work, volume fractions of bagasse fibers (10, 20%, 30% and 40% by weight) have been taken as reinforcement in the polymer matrix.

3.1.2 Epoxy resin

Softener (Araldite LY 556) made by CIBA GEIGY limited having the following outstanding properties has been used as the matrix material.

a. Excellent adhesion to different materials.

b. High resistance to chemical and atmospheric attack.
c. High dimensional stability.
d. Free from internal stresses.
e. Excellent mechanical and electrical properties.
f. Odourless, tasteless and completely nontoxic.
g. Negligible shrinkage.

3.1.3 Hardener

In the present work hardener (HY951) is used. This has a viscosity of 10-20 MPa at 25ºc.

3.2 PREPARATION OF COMPOSITES:

The following procedure has been adopted for the preparation of the specimen.

(a) Bagasse fiber preparation

Fresh bagasse fibers were collected from Sakti Sugar Industries located at Dhenkanal, Orissa. These fibers were then spread on a water proof sheet and stored in an enclosed shed to reduce the moisture content. After approximately two weeks, the long bagasse fibers (rind portion only) were shortened into a length of 12mm to 15mm, breadth of 1mm and width of 1mm with a pair of scissors. Small size fibers were selected in order to design a composite with consistent properties. Due to the low moisture content of the bagasse samples, no fungi grew during the storage. The bagasse samples were then cleaned via pressurized water for about one hour. This procedure removes fine bagasse particles, sugar residues and organic materials from the samples. Then the fibers were dried with compressed air at a pressure of approximately 145kPa at 108^0C. The required drying time was determined by weighing a trial sample every ten minutes until the measured mass becomes constant. A drying time of 40 min was established to provide sufficient drying of the fiber. Fig 3.1 shows SEM micrographs of the cross section of a bagasse fiber.

b) Evaluation of critical fiber length

Since we are going to use short, non-continuous fibers in composite it is essential to determine the critical fiber length. Standard pullout test was carried out for the purpose .This test provides useful information about the interface strength in model composite system. One must also carefully avoid any fiber misalignment and introduction of

bending moments. Figure 3.5 shows a schematic of the experimental assemblage. Pull out tests were performed using embedded fiber lengths L, varying from 3 to 20mm.The single bagasse fiber were mounted on epoxy sockets .The pullout tests were performed on a UTM machine with 10 KN capacity with a crosshead speed of 1mm/min. Figure 3.6 shows the samples tested. The free fiber end was clamped with pneumatic action grips. The critical fiber length found out experimentally is 10mm.

(b) Composite preparation

A wooden mold of dimension (120x100x6) mm was used for casting the composite sheet. The first group of samples was manufactured with 10, 20, 30, and 40 % volume fraction of fibers**.** Usual hand lay-up technique was used for preparation of the samples. For different volume fraction of fibers, a calculated amount of epoxy resin and hardener (ratio of 10:1 by weight) was thoroughly mixed with gentle stirring to minimize air entrapment. For quick and easy removal of composite sheets, mold release sheet was put over the glass plate and a mold release spray was applied at the inner surface of the mold. After keeping the mold on a glass sheet a thin layer (≈ 2 mm thickness) of the mixture was poured. Then the required amount of fibers was distributed on the mixture. The remainder of the mixture was then poured into the mold. Care was taken to avoid formation of air bubbles. Pressure was then applied from the top and the mold was allowed to cure at room temperature for 72 hrs. This procedure was adopted for preparation of 10, 20, 30 and 40% fiber volume fractions of composites. After 72 hrs the samples were taken out of the mold, cut into different sizes and kept in air tight container for further experimentation.

3.3 ENVIRONMENTAL TREATMENT

To find out the effect of environment on mechanical properties the composite samples were subjected to various treatments like:

(a) Steam treatment
(b) Saline treatment
(c) Subzero condition

In each conditions a set of composites (10, 20, 30, 40% volume fraction) were tested for various time lengths. Steam treatment was conducted at 100^0C with 95 % relative humidity. Subzero treatment was conducted at -23^0C and saline treatment was done with 5%

concentration. At the end of the treatment in each condition, the dimensions and weight change were measured.

3.4 MOISTURE ABSORPTION

The water absorption experiment was carried out according to the following procedure. The samples were oven dried at 50°C for 24hrs to a constant weight (M_0). Then the weighed specimens were exposed to different environmental conditions (steam, saline and sub zero). The samples were then periodically (after each 8hrs) taken out from the conditions. The surface was dried with absorbent paper and weighed immediately to determine the wet weight of the specimens (M_1). The percentage weight gain at any time t (M_t) as the result of water absorption was determined by using the following equation.

$$\mathbf{M_t\ (\%) = [(M_1 - M_0)/M_0]\ X100}$$

Where M_0 is the dry initial weight and M_1 is the weight after exposure to different environmental conditions.

3.5 FLEXURAL STRENGTH

Three point bend test was carried out in an UTM 201 machine in accordance with ASTM D2344-84 to measure the flexural strength of the composites after subjecting them in various weathering conditions continuously for 64 hrs. The loading arrangement for the specimen and the photograph of the machine used are shown in fig 3.2 & fig 3.3 All the specimens (composites) were of rectangular shape having length varied from 100-125 mm, breadth of 100-110 mm and thickness of 4-6 mm. A span of 100 mm was employed maintaining a cross head speed of 10mm/min.

The flexural inter laminar shear strength (ILSS) of the composite which is the maximum shear stress that a material can withstand before it ruptures, was calculated using the equation

$$\sigma\ m = 3f/4bt$$

Where σm is the ILSS, f is the load, b is the width and t is the thickness of the specimen under test. The maximum tensile stress was found out form the equation.

$$\tau m = 3fl/2bt^2$$

Where τm is the maximum tensile stress and l is the gauge length.

The results obtained are tabulated in table no 3.1

Fig 3.4 shows the variation in flexural strength for the composite in natural, steam, saline and subzero environment. The plot shows that, the samples with 30% fiber volume fraction gives maximum strength in normal condition and also in steam, saline and subzero conditions. So for further experimentation 30% fiber volume fraction of composite has been taken.

Table- 3.1

Flexural Strength & shear stress of unwashed fiber in different condition of treatment

Types of Composites	Conditions of Treatment	**10%**		**20%**		**30%**		**40%**	
		Shear Stress (MPa)	Flexural Strength (MPa)	Shear Stress (MPa)	Flexural Strength (MPa)	Shear Stress (MPa)	Flexural Strength (MPa)	Shear Stress (MPa)	Flexural Strength (MPa)
Unwashed	Normal	1.387	6.98	0.278	9.952	0.582	20.98	0.511	14.17
	Steam	0.556	4.24	0.208	6.426	0.284	9.512	0.310	8.524
	Saline	0.764	5.26	0.266	8.885	0.499	17.52	0.349	9.699
	Subzero	0.862	6.12	0.318	10.56	0.370	10.09	0.513	14.60

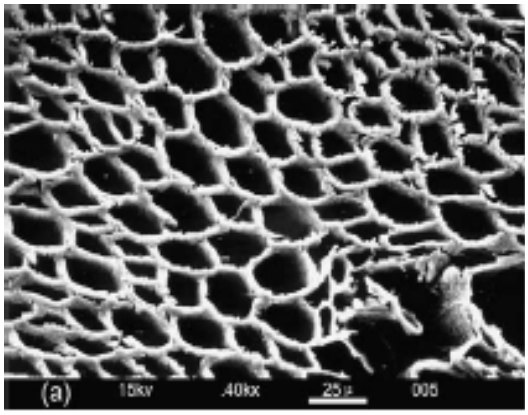


Fig 3.1 SEM micrographs of the cross section of a bagasse fiber.

Fig 3.2 Testing Machine

Fig 3.3 Testing machine with the specimen in loading position

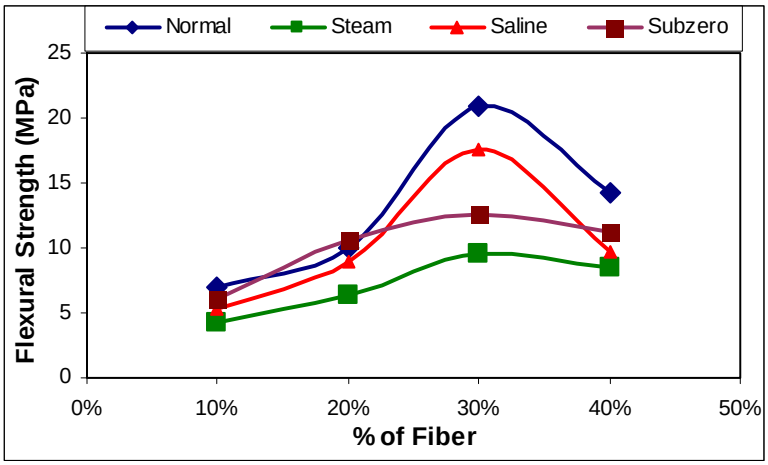


Fig 3.4 Flexural strength of 10, 20, 30 and 40% fiber volume Fraction of composites under normal steam, saline and subzero conditions

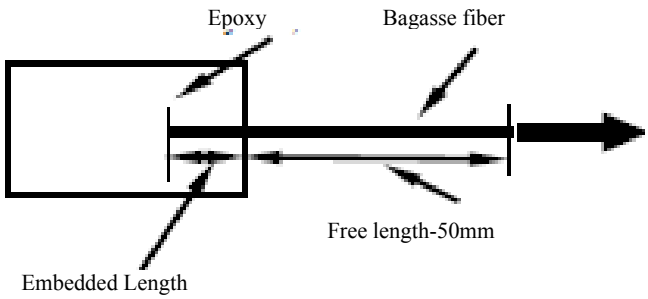


Fig 3.5 Schematic representation of pull out test

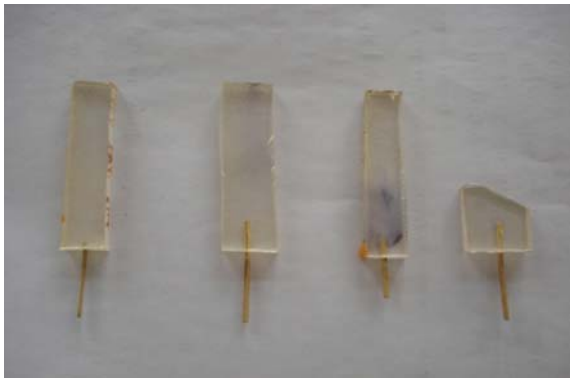


Fig 3.6 Specimen for pullout test

Chapter 4

EFFECT OF CHEMICAL MODIFICATION ON MECHANICAL PROPERTIES OF BAGASSE FIBER COMPOSITE

CHAPTER – 4

4.1 INTRODUCTION

It is concluded from the previous chapter that the strength of the composites increases with increase in fiber volume fraction when subjected to different environmental treatment. Since the interfacial bonding between the reinforcing fibers and the resin matrix is an important element in realizing the mechanical properties, several authors [45-48] have focused the studies on the treatment of fibers to improve the bonding with resin matrix. The mechanical properties of the composites are controlled by the properties and quantities of the component materials and by the character of the interfacial region between matrix and reinforcement. Lack of good interfacial adhesion makes the use of cellular fiber composites less attractive. Often the low interfacial properties between the fiber and polymer matrix, because of hydrophilic nature of natural fiber reduces the potential of being used as reinforcing agents. Chemical modifications are considered to optimize the interface of fibers. Chemicals may activate hydroxyl groups or introduce new moieties that can effectively interlock with the matrix. There are various chemical treatments available for the fiber surface modification. Chemical treatment including alkali, silane, acetylation, benzoylation, acrylation, isocynates, maleated coupling agents, permanganate treatment are discussed in details in [49].

The chemical treatment of fiber aimed at improving the adhesion between the fiber surface and the polymer matrix may not only modify the fiber surface but also increase the fiber strength. Water absorption of composites is reduced and their mechanical properties are improved. Out of the available treatments, for the present case to have a good bonding between the fiber and the resin matrix bagasse have been treated with alkali & acetone. The subsequent section will elaborate separately the treatment of the fiber surface by these two methods, weathering behaviour, measurement of flexural strength and result obtained to achieve the objective.

4.2 FIBER MODIFICATION

a) Alkaline Treatment

Alkaline treatment or mercerization is one of the most used chemical treatments of natural fibers when used to reinforce thermoplastics and thermosets. The important modification

done by alkaline treatment is the disruption of hydrogen bonding in the network structure, thereby increasing surface roughness. This treatment removes a certain amount of lignin, wax and oils covering the external surface of the fiber cell wall, depolymerizes cellulose and exposes the short length crystallites [50]. Addition of aqueous sodium hydroxide (NaOH) to natural fiber promotes the ionization of the hydroxyl group to the alkoxide [51].

$$\textbf{Fiber} - \textbf{OH} + \textbf{NaOH} \rightarrow \textbf{Fiber} - \textbf{O} - \textbf{Na} + \textbf{H}_2\textbf{O}$$

Thus, alkaline processing directly influences the cellulosic fibrill, the degree of polymerization and the extraction of lignin and hemicellulosic compounds [52]. It is reported that alkaline treatment has two effects on the fiber:

1) It increases surface roughness resulting in better mechanical interlocking, and
2) It increases the amount of cellulose exposed on the fiber surface, thus increasing the number of possible reaction sites [53].

Consequently, alkaline treatment has a lasting effect on the mechanical behaviour of flax fiber, especially on fiber strength and stiffness.

b) Alkali treatment of bagasse fibers

The effect of alkali treatments (0%, 1%, 3%,5%) of bagasse fibers on the flexural strengths were examined using treated fiber composites. As seen from figure 4.1, the maximum improvement in the flexural strength of the composite was observed for 5% NaOH treated fiber composites. It was believed that better interfacial adhesion along with better fibrillation of these fibers contributed effectively to the enhancement in the flexural properties.

For treatment in alkali the bagasse fibers were soaked in a 5% NaOH solution at room temperature maintaining a liquor ratio of 15:1. The fibers were kept immersed in the alkali solution for 2, 4 and 6hrs. The fibers were then washed several times with fresh water to remove any NaOH sticking to the fiber surface, neutralize with dilute acetic acid and finally washed again with distilled water. A final pH of 7 was maintained. The fibers were then dried at room temperature for 48 hrs followed by oven drying at 100°C for 6hrs. The alkali reaction between bagasse fiber and NaOH is as follows:

$$Bagasse - OH + NaOH \Leftrightarrow Bagasse - O^{-}Na^{+} + H_2O$$

The NaOH reacts with hydroxyl groups of the cementing material hemicellulose, and it brings on the destruction of the cellular structure and thereby the fibers split into filaments. The difference between the fiber before and after alkali treatments is exhibited in fig 4.2. It was observed that the filaments in the untreated fiber were packed together but got split after the alkali treatment. This phenomenon is termed as fibrillation, which breaks the untreated fiber bundle down into smaller ones by the dissolution of the hemicellulose. The fibrillation increases effective surface area available for contact with the matrix [54] and hence the interfacial adhesion was improved.

c) Acetone Treatment

When the fiber is treated with acetone, the lignin, cellulolignin and other such material get dissolved in acetone. As acetone is a non-polar organic solvent it usually dissolves the non-polar organic component.

d) Acetone treatment of bagasse fibers

The bagasse fibers were washed in soxhlet extractor (fig 4.3) with acetone for approximately 1-1.5hrs. The acetone was evaporated (boiled at 63^0C) and condensed back into the volume with the fibers. This process was repeated four times for each batch. The used acetone was discarded before the new batch was cleaned in the same manner. The acetone changed from transparent to bottle green after treatment due to the presence of waxes and organic materials after the extraction. All the fibers were washed with pressurized water at a temperature of 90^0C for 70 minutes before acetone treatment except unwashed fibers.

In the present investigation the fibers were treated with acetone and the groups of samples involved are 30 % fiber volume fraction of varying bagasse fiber surface treatment, namely unwashed, unwashed treated with acetone and washed treated with acetone composites.

4.3 EXPERIMENTAL PROCEDURE

After the preparation of fibers by alkali and acetone, they were used for preparation of samples. The preparation of samples is same as described in chapter 3 of art 3.2. The only difference is that we have taken only 30% volume fraction of fibers which is found to be optimum. The experimental procedure for finding out the effect of environment and flexural strength for the treated fibers is also same as explained in chapter 3 of art 3.2. Only the results

found out for the treated fibers in comparision to the untreated fibers are explained in the subsequent sections.

4.4 RESULTS AND DISCUSSIONS

4.4.1 Alkali treatment

The results of prepared samples (volume change, weight change and flexural strength) subjected to different environmental conditions are presented in table no. 4.1 to 4.7

Fig 4.4 (a-d) shows the flexural strength of the composite for unwashed treated and washed treated fibers with alkali for 2, 4 and 6 hours after subjecting them to different environmental condition. It is evident from Figure that in all cases the flexural strength is maximum for both washed and 4 hrs treated fiber composites. The increase in flexural strength is about 7.67%, 23.34%, 17.43% and 17.56% for normal, steam, saline and subzero conditions respectively. Hence for rest of the investigations composites were prepared with 4 hours alkali treated fibers for analysis.

Fig 4.5 to 4.7 shows the cumulative volume change for alkali treated, 30% volume fraction unwashed, washed treated and unwashed treated reinforced fibers subjected to steam, saline and subzero.

It is seen from the plot (fig 4.5); the change in volume for washed treated fiber is minimum. The washed treated and unwashed treated samples get stabilized at an early exposure time in comparison to unwashed samples. It is also seen that the volume change is much higher for unwashed samples. This may due to swelling of fibers. This behavior is attributed to the hygroscopic nature of the fibers. When the fibers were treated, this property gets decreased and hence less swelling of the fibers when subjected to steam.

Fig 4.6 shows volume change of the samples subjected to saline water. The washed treated and unwashed treated samples get stabilized after 24 hours of treatment whereas the unwashed samples show an indication of saturation at about 56 hours of treatment. This early stabilization for washed treated and unwashed treated samples is due to the formation of mono layers which formed due to electron rich species with sodium ions. It is interesting to note that beyond 40 hours of treatment the swelling in the composite for washed treated and

unwashed treated suddenly increases to a higher value. It can be concluded here for this type of behavior that after forming a mono layer of sodium ions in the surface it is attracting the opposite charge ions which achieves equilibrium after 40 hours and forms a double layer. The double layer thus formed is responsible for increase in the volume after 40 hours.

Fig 4.7 shows the change in volume under subzero treatment for the composite. A large variation in magnitude for the change in volume is seen during the treatment period for all the composite. However this variation is less for the washed treated and unwashed treated composite. The washed treated and unwashed treated samples as it is seen from the plot may go towards stabilization after 56 hours of treatment. This type of behavior of the composite for subzero treatment may be due to less intermolecular hydrogen bonding.

Fig 4.8 to 4.10 shows the percentage change in weight for different time of exposure under varying environmental condition for the composite. All these curves shows similar trends but with variation in magnitude (of weight). About 24 hours of exposure in all the treatments the linearity in the curve is achieved for washed treated samples where as it takes longer time for unwashed and unwashed treated samples. From this it can be concluded that treating the fiber with alkali has a positive effect.

Fig 4.11 shows the flexural strength of unwashed, washed treated and unwashed treated composites samples. It is clear that flexural strength of washed and 4 hrs alkali treated composites is maximum for all cases as compared to unwashed and unwashed treated fiber composites.

Fig 4.12 shows the shear stress of the composite under different environmental treatment. It is clear from the plot that shear stress of washed and 4 hrs alkali treated composites is maximum for all cases as compared to unwashed and unwashed treated fiber composites. There is a large variation on the shear stress values when the composite is subjected to steam and saline treatment ,whereas it is almost negligible for normal and subzero condition.

4.4.1.1 Fractographic Analysis

Fig 4.13 (a) shows that most of the fibers have come out without breaking during fracture for the composite subjected to steam treatment. This might have occurred due to dissolution of cellulose constituent in alkali which creates voids in the fiber structure,

increases swelling and makes the fiber weaker. Destruction of mess network and splitting of fibers in to filaments that might have occurred during treatment. Fig 4.13 (b) also shows breaking down of fiber bundles into smaller one. This increases the effective surface area available for wetting by the resin and when subjected to subzero conditions, the absorption of water as explained earlier is less hence indicates higher flexural strength. Fig 4.13 (c) shows the micrograph for saline exposed sample. Same type of features are seen as for subzero condition. The breaking of fibers due to fibrillation is clearly visible hence higher strength.

4.4.2 Acetone treatment

After exposing the composites to various environmental conditions viz. steam, saline and subzero treatments, the changes in the different properties are evaluated. The results are tabulated in table 4.8 to 4.14.

The results of steam swelling and steam absorption are shown in fig 4.14 and 4.15 respectively. It is observed from the results that the linearity in the curves is achieved at about 32 hours for all cases. It is also observed that washed and treated bagasse samples exhibited the least swelling among all.

Absorption of steam (fig 4.15) increases at a faster rate up to 40 hrs, for unwashed samples and then got stabilized.. How ever the rate of absorption of moisture is much slower for the washed treated and unwashed treated samples with varying moisture absorption and least swelling is observed for the washed treated samples.

During saline treatment (fig 4.16 and fig 4.17) not only moisture absorption takes place but also transport of sodium and chlorine ions do occur leading to some what a chemical reaction with the matrix as well as with the fiber. Therefore the swelling is much higher for unwashed fibers, where as the deviation is not so much for the washed treated and unwashed treated fiber. Least swelling is observed for washed treated fiber.

Fig 4.19 shows the trend in water absorption from 8 hrs to 64 hrs while fig 4.18 represents the water swelling for subzero treatment. The rate of absorption of water is linear in all the cases after 48 hrs while for washed treated samples it shows linearity after 40 hrs. The trend in water absorption is washed treated < unwashed < unwashed treated.

The water swelling in washed treated samples is the lowest. It is seen from the plot (fig 4.18) that unwashed fiber and the unwashed treated fiber sample lie near to each other. There is dramatic shift for washed treated samples which can be visualized from the plot.

There is little difference in water absorption for washed treated samples with respect to unwashed samples. This is due to the spongy nature of the pith of the bagasse which can absorb more water; but the swelling for washed treated samples is much lower because of removal of lignin content in the surface of fibers and fibrils with the acetone.

The variations in flexural strength of the composites under various environmental conditions are presented in fig 4.20. It is clear from the plot that the washed and treated fiber composites posses the maximum strength in all the environmental conditions viz. natural, steam, saline and subzero treatment. The flexural strength of the washed treated fiber composites increases by about 30- 50 % of the unwashed, unwashed treated fiber composites in all the treatments. This improved property of the composite is due to the treatment of fiber with acetone which results in dissolution of hemicellulose, development of crystallinity and fibrillation thus creating superior bonding with matrix.

Fig 4.21 shows the variation in shear stress of the composites under different environmental conditions. The plot shows that the washed and treated fiber composites shows maximum value in all the environmental conditions they are subjected to.

4.4.2.1 Study of failure modes

The composites processed after treating the fiber in acetone has improved the strength properties. The fracture surfaces after exposed to different environment are shown in fig 4.22 (a-c).

Fig. 4.22 (a) shows the fiber breakage instead of pull out of fibers from the matrix. It also indicates that cellulose structure has been compressed but not so much as to prevent fracture of some fibers. The breakage of fibers indicates better interfacial strength. Fig 4.22 (b) shows the morphology of the samples subjected to subzero condition. There is no trace of fiber breakage in the composite, which indicates good bonding between fiber and the matrix. Swelling of fiber is less hence higher strength. When the composite is subjected to saline environment it indicates flexural strength comparable to subzero environment. Probably, Fig 4.22 (c) the fiber matrix bonding has been improved due to formation of monolayer which controls the propagation of moisture through fibril interfaces.

4.5 CONCLUSIONS

The following conclusions are drawn from the present work.

1. The sugar cane residue bagasse an underutilized renewable agricultural material can successfully be utilized to produce composite by suitably bonding with resin for value added product.

2. By comparing the flexural strength of the composites with varying fiber treatment, the best mechanical property results are obtained with bagasse fiber that are both washed and treated with either alkali or acetone.

3. Fibrillation in the fiber bundles has taken place when the fibers were treated with alkali by the dissolution of hemicellulose.This also increases the effective suface area available for contact with the matrix and hence the interfacial adhesion was improved. Acetone treatment increases the property of the fibers by dissolution of hemi cellulose thus creating a superior bonding with the matrix.

4. Between 2, 4 and 6 hrs alkali treated fiber composites, the washed and 4hr alkali treated fiber composites shows maximum flexural strength than the unwashed treated composites.

5. From the morphology of the fractured surface (treated under different environment) for the alkali treated fiber it was found that fiber breakage were the predominant mode of failure.

6. For acetone treatment, fibers pullout were the predominant mode of failure. Therefore the strength in acetone treated fiber is some what low in comparison to alkali treated fiber. However it is established that fiber matrix bonding has improved a lot by chemical modification in comparison the untreated fibers.

Table -4.1

Cumulative Volume Change for 30% 4 hours (NaOH Treated) fiber volume fraction composites in steam treatment

Types on composites	Unwashed Volume (cm^3)			Washed Treated Volume (cm^3)			Unwashed Treated Volume (cm^3)		
Treatment (Hrs)	Initial	Final	Diff	Initial	Final	Diff	Initial	Final	Diff
8	14.85	16.74	1.89	14.84	15.91	1.07	11.66	13.09	1.43
16	14.85	16.64	1.79	14.84	16.51	1.67	11.66	12.92	1.26
24	14.85	16.12	1.27	14.84	15.80	0.96	11.66	12.42	0.76
32	14.85	16.42	1.57	14.84	15.91	1.07	11.66	12.67	1.01
40	14.85	16.70	1.85	14.84	15.78	0.94	11.66	12.85	1.19
48	14.85	16.33	1.48	14.84	15.83	0.98	11.66	12.84	1.18
56	14.85	16.42	1.57	14.84	15.88	1.04	11.66	12.68	1.02
64	14.85	16.47	1.62	14.84	15.73	0.89	11.66	12.72	1.06

Table -4.2

Cumulative Volume Change for 30 % 4 hours (NaOH Treated) fiber volume fraction composites in saline treatment

Types on composites	Unwashed Volume (cm^3)			Washed Treated Volume (cm^3)			Unwashed Treated Volume (cm^3)		
Treatment (Hrs)	Initial	Final	Diff	Initial	Final	Diff	Initial	Final	Diff
8	12.78	12.87	0.09	13.52	14.29	0.77	13.71	14.79	1.08
16	12.78	13.05	0.27	13.52	13.95	0.43	13.71	14.2	0.49
24	12.78	13.16	0.38	13.52	13.76	0.24	13.71	14.25	0.54
32	12.78	13.32	0.54	13.52	13.80	0.28	13.71	14.32	0.61
40	12.78	13.23	0.45	13.52	13.82	0.30	13.71	14.15	0.44
48	12.78	13.79	1.01	13.52	14.18	0.66	13.71	14.72	1.01
56	12.78	13.41	0.63	13.52	14.60	1.08	13.71	14.78	1.07
64	12.78	13.50	0.72	13.52	14.77	1.25	13.71	14.95	1.24

Table -4.3

Cumulative Volume Change for 30 % 4 hours (NaOH Treated) fiber volume fraction composites in subzero treatment

Types on composites	Unwashed Volume (cm^3)			Washed Treated Volume (cm^3)			Unwashed Treated Volume (cm^3)		
Treatment (Hrs)	Initial	Final	Diff	Initial	Final	Diff	Initial	Final	Diff
8	16.06	16.08	0.02	12.67	12.75	0.08	12.77	12.8	0.03
16	16.06	16.49	0.43	12.67	13.01	0.34	12.77	13.01	0.24
24	16.06	16.95	0.89	12.67	13.02	0.35	12.77	13.21	0.44
32	16.06	16.20	0.14	12.67	13.18	0.51	12.77	13.33	0.56
40	16.06	16.28	0.22	12.67	12.96	0.29	12.77	12.98	0.21
48	16.06	16.50	0.44	12.67	13.13	0.46	12.77	13.46	0.69
56	16.06	16.67	0.61	12.67	12.96	0.29	12.77	12.94	0.17
64	16.06	16.19	0.13	12.67	13.00	0.33	12.77	12.90	0.13

Table -4.4

Cumulative Weight Change for 30 % 4 hours (NaOH Treated) fiber volume fraction composites in steam treatment

Types on composites	Unwashed Weight (gram)			Washed Treated Weight (gram)			Unwashed Treated Weight (gram)		
Treatment (Hrs)	Initial	Final	Diff	Initial	Final	Diff	Initial	Final	Diff
8	15.01	16.22	1.21	15.53	16.00	0.47	12.87	13.52	0.65
16	15.01	16.54	1.53	15.53	16.17	0.64	12.87	13.71	0.84
24	15.01	17.07	2.06	15.53	16.30	0.77	12.87	13.91	1.04
32	15.01	16.95	1.94	15.53	16.30	0.77	12.87	13.98	1.11
40	15.01	17.13	2.12	15.53	16.39	0.86	12.87	14.00	1.13
48	15.01	17.07	2.06	15.53	16.40	0.87	12.87	14.11	1.24
56	15.01	17.15	2.14	15.53	16.42	0.89	12.87	14.02	1.15
64	15.01	17.31	2.30	15.53	16.42	0.89	12.87	13.98	1.11

Table -4.5

Cumulative Weight Change for 30 % 4 hours (NaOH Treated) fiber volume fraction composites in saline treatment

Types on composites	Unwashed Weight (gram)			Washed Treated Weight (gram)			Unwashed Treated Weight (gram)		
Treatment (Hrs)	Initial	Final	Diff	Initial	Final	Diff	Initial	Final	Diff
8	13.05	13.44	0.39	14.51	14.59	0.08	14.54	14.65	0.11
16	13.05	13.73	0.68	14.51	14.76	0.25	14.54	14.71	0.17
24	13.05	13.61	0.56	14.51	14.73	0.22	14.54	14.72	0.18
32	13.05	13.72	0.67	14.51	14.69	0.18	14.54	14.72	0.18
40	13.05	13.69	0.64	14.51	14.74	0.23	14.54	14.75	0.21
48	13.05	13.89	0.84	14.51	14.81	0.30	14.54	14.87	0.33
56	13.05	14.00	0.95	14.51	14.75	0.24	14.54	14.79	0.25
64	13.05	14.12	1.07	14.51	14.89	0.38	14.54	14.91	0.37

Table -4.6

Cumulative Weight Change for 30 % 4 hours (NaOH Treated) fiber volume fraction composites in subzero treatment

Types on composites	Unwashed Weight (gram)			Washed Treated Weight (gram)			Unwashed Treated Weight (gram)		
Treatment (Hrs)	Initial	Final	Diff	Initial	Final	Diff	Initial	Final	Diff
8	14.46	14.48	0.02	13.61	13.62	0.01	14.85	14.87	0.02
16	14.46	14.59	0.13	13.61	13.63	0.02	14.85	14.86	0.01
24	14.46	14.50	0.04	13.61	13.64	0.03	14.85	14.86	0.01
32	14.46	14.56	0.10	13.61	13.64	0.03	14.85	14.92	0.07
40	14.46	14.54	0.08	13.61	13.70	0.09	14.85	14.99	0.14
48	14.46	14.50	0.04	13.61	13.64	0.03	14.85	14.88	0.03
56	14.46	14.55	0.09	13.61	13.64	0.03	14.85	14.92	0.07
64	14.46	14.55	0.09	13.61	13.64	0.03	14.85	14.91	0.06

Table-4.7

Flexural strength of NaOH treated fiber composites

Types of Composites	Conditions of Treatment	30%	
		Shear Stress (MPa)	**Flexural Strength (MPa)**
Unwashed	Normal	0.582	20.98
	Steam	0.284	9.512
	Saline	0.499	17.52
	Subzero	0.370	10.09
Washed Treated (4 Hours)	Normal	0.642	22.58
	Steam	0.416	15.48
	Saline	0.721	25.26
	Subzero	0.680	28.71
Unwashed Treated (4 Hours)	Normal	0.492	20.97
	Steam	0.264	10.71
	Saline	0.614	21.51
	Subzero	0.655	24.40

Table 4.8

Cumulative volume change of acetone treated fiber composite for steam treatment

Types of Composites	Unwashed			Washed Treated			Unwashed Treated		
Treatment (Hrs)	Volume (cm^3)			Volume (cm^3)			Volume (cm^3)		
	Initial	Final	Diff	Initial	Final	Diff	Initial	Final	Diff
8	14.85	16.74	1.89	13.8	13.98	0.18	13.37	14.12	0.75
16	14.85	16.64	1.79	13.8	14.16	0.36	13.37	14.1	0.73
24	14.85	16.12	1.27	13.8	14.21	0.41	13.37	14.26	0.89
32	14.85	16.42	1.57	13.8	14.39	0.59	13.37	14.34	0.97
40	14.85	16.44	1.59	13.8	14.32	0.52	13.37	14.33	0.96
48	14.85	16.48	1.63	13.8	14.28	0.48	13.37	14.35	0.98
56	14.85	16.47	1.62	13.8	14.28	0.48	13.37	14.33	0.96
64	14.85	16.47	1.62	13.8	14.28	0.48	13.37	14.33	0.96

Table 4.9

Cumulative volume change of acetone treated fiber composite for saline treatment

Types of Composites	Unwashed			Washed Treated			Unwashed Treated		
Treatment (Hrs)	Volume (cm^3)			Volume (cm^3)			Volume (cm^3)		
	Initial	Final	Diff	Initial	Final	Diff	Initial	Final	Diff
8	12.78	12.87	0.09	13.39	13.51	0.12	14.20	14.40	0.20
16	12.78	13.05	0.27	13.39	13.64	0.25	14.20	14.42	0.22
24	12.78	13.16	0.38	13.39	13.56	0.17	14.20	14.30	0.10
32	12.78	13.32	0.54	13.39	13.46	0.07	14.20	14.30	0.10
40	12.78	13.39	0.61	13.39	13.46	0.07	14.20	14.42	0.22
48	12.78	13.39	0.61	13.39	13.46	0.07	14.20	14.43	0.23
56	12.78	13.38	0.60	13.39	13.55	0.06	14.20	14.43	0.23
64	12.78	13.38	0.60	13.39	13.55	0.06	14.20	14.43	0.23

Table 4.10

Cumulative volume change of acetone treated fiber composite for subzero treatment

Types of Composites	Unwashed			Washed Treated			Unwashed Treated		
Treatment (Hrs)	Volume (cm^3)			Volume (cm^3)			Volume (cm^3)		
	Initial	Final	Diff	Initial	Final	Diff	Initial	Final	Diff
8	16.06	16.08	0.02	14.17	14.40	0.23	13.57	13.66	0.09
16	16.06	16.49	0.43	14.17	14.57	0.40	13.57	13.69	0.12
24	16.06	16.95	0.89	14.17	14.69	0.52	13.57	13.72	0.15
32	16.06	16.20	0.14	14.17	14.74	0.57	13.57	14.01	0.44
40	16.06	16.28	0.22	14.17	14.39	0.22	13.57	13.91	0.34
48	16.06	16.50	0.44	14.17	14.41	0.24	13.57	14.14	0.57
56	16.06	16.48	0.42	14.17	14.41	0.24	13.57	14.11	0.54
64	16.06	16.47	0.41	14.17	14.41	0.24	13.57	14.11	0.54

Table 4.11

Cumulative weight change of acetone treated fiber composite for steam treatment

Types of Composites	Unwashed			Washed Treated			Unwashed Treated		
Treatment (Hrs)	Weight (gm)			Weight (gm)			Weight (gm)		
	Initial	Final	Diff	Initial	Final	Diff	Initial	Final	Diff
8	15.01	16.22	1.21	13.30	14.1	0.80	13.14	14.18	1.04
16	15.01	16.54	1.53	13.30	14.45	1.15	13.14	14.48	1.34
24	15.01	17.07	2.06	13.30	15.00	1.70	13.14	14.89	1.75
32	15.01	16.95	1.94	13.3	15.19	1.89	13.14	15.07	1.93
40	15.01	17.13	2.12	13.30	15.21	1.91	13.14	15.05	1.91
48	15.01	17.07	2.06	13.30	15.32	2.02	13.14	15.18	2.04
56	15.01	17.06	2.05	13.30	15.31	2.01	13.14	15.17	2.03
64	15.01	17.06	2.05	13.30	15.31	2.01	13.14	15.17	2.03

Table 4.12

Cumulative weight change of acetone treated fiber composite for saline treatment

Types of Composites	Unwashed			Washed Treated			Unwashed Treated		
Treatment (Hrs)	Weight (gm)			Weight (gm)			Weight (gm)		
	Initial	Final	Diff	Initial	Final	Diff	Initial	Final	Diff
8	13.05	13.44	0.39	14.34	14.57	0.23	13.18	13.46	0.28
16	13.05	13.73	0.68	14.34	14.69	0.35	13.18	13.59	0.41
24	13.05	13.61	0.56	14.34	14.75	0.41	13.18	13.67	0.49
32	13.05	13.72	0.67	14.34	15.02	0.68	13.18	13.91	0.73
40	13.05	13.69	0.64	14.34	15.07	0.73	13.18	13.96	0.78
48	13.05	13.89	0.84	14.34	15.12	0.78	13.18	14.03	0.85
56	13.05	13.88	0.83	14.34	15.12	0.78	13.18	14.03	0.85
64	13.05	13.88	0.83	14.34	15.12	0.78	13.18	14.03	0.85

Table 4.13

Cumulative weight change of acetone treated fiber composite for subzero treatment

Types of Composites	Unwashed			Washed Treated			Unwashed Treated		
Treatment (Hrs)	Weight (gm)			Weight (gm)			Weight (gm)		
	Initial	Final	Diff	Initial	Final	Diff	Initial	Final	Diff
8	13.46	13.58	0.12	14.46	14.48	0.02	14.05	14.17	0.12
16	13.46	13.78	0.32	14.46	14.59	0.13	14.05	14.34	0.29
24	13.46	13.76	0.30	14.46	14.5	0.04	14.05	14.36	0.31
32	13.46	13.75	0.29	14.46	14.56	0.10	14.05	14.34	0.29
40	13.46	13.75	0.29	14.46	14.54	0.08	14.05	14.33	0.28
48	13.46	13.73	0.27	14.46	14.55	0.09	14.05	14.32	0.27
56	13.46	13.73	0.27	14.46	14.55	0.09	14.05	14.33	0.28
64	13.46	13.73	0.27	14.46	14.55	0.09	14.05	14.32	0.27

Table 4.14

Flexural Strength of acetone treated fiber composites

Types of Composites	Conditions of Treatment	Shear Stress (MPa)	Flexural Stress (MPa)
Unwashed	Normal	0.408	20.98
	Steam	0.284	9.512
	Saline	0.499	17.52
	Subzero	0.370	10.09
Washed Treated	Normal	0.495	22.26
	Steam	0.385	16.85
	Saline	0.619	25.14
	Subzero	0.626	26.74
Unwashed Treated	Normal	0.432	17.28
	Steam	0.341	14.19
	Saline	0.459	20.40
	Subzero	0.589	26.37

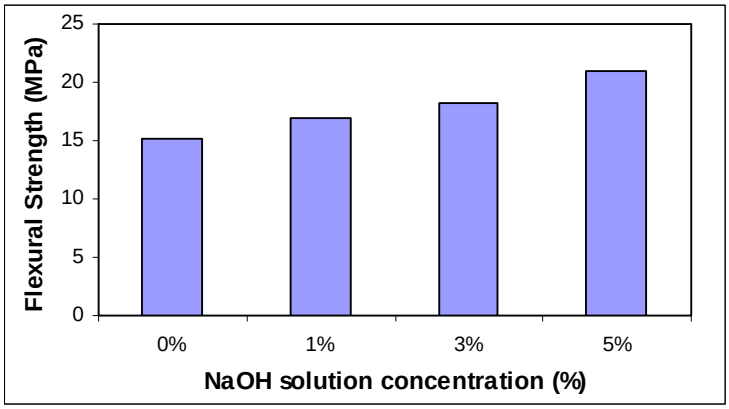
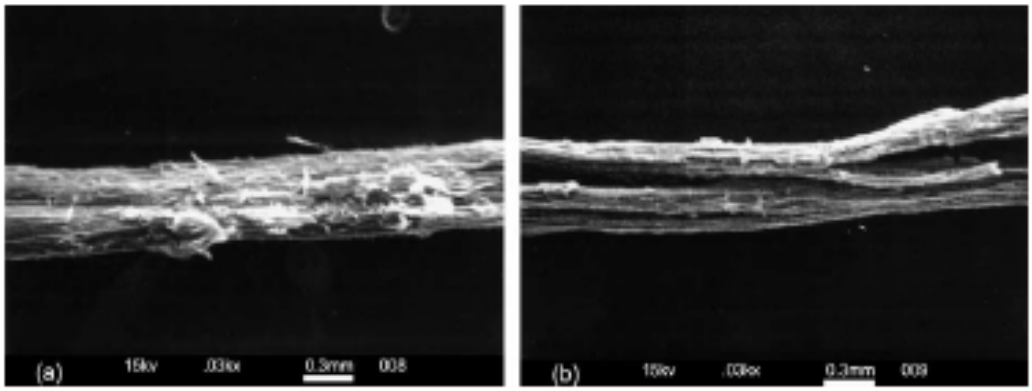


Fig 4.1 Effect of alkali treatment on mechanical properties of composites



Untreated fiber **5% NaOH treated fiber**

Fig 4.2 SEM micrograph of a baggsse fiber

Fig 4.3 Soxhlet Extractor

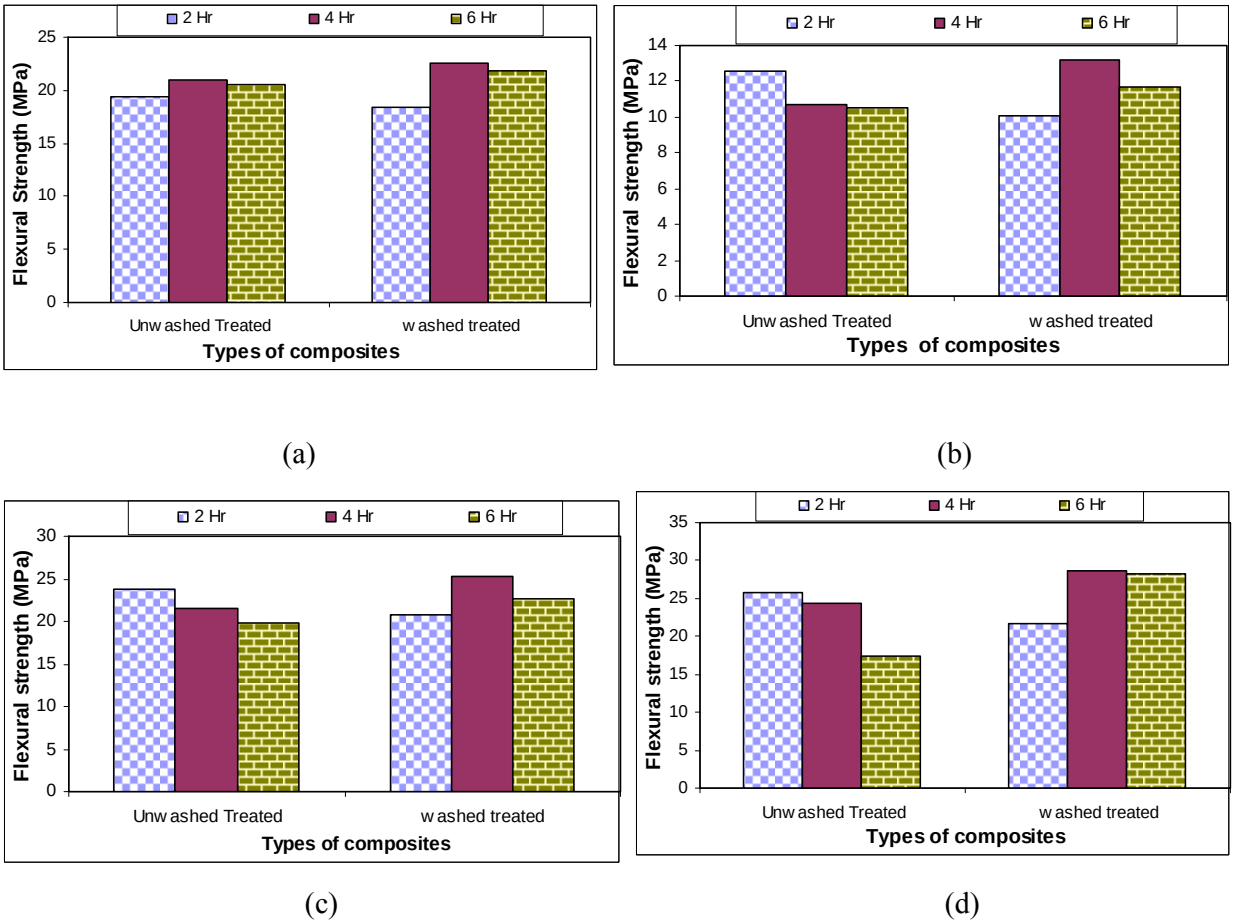


Fig. 4.4 (a-d) Flexural strength of 30% volume fraction of (unwashed and washed) 2, 4 and 6 hrs of alkali treated fiber composites under a)normal b)steam c)saline d) subzero conditions.

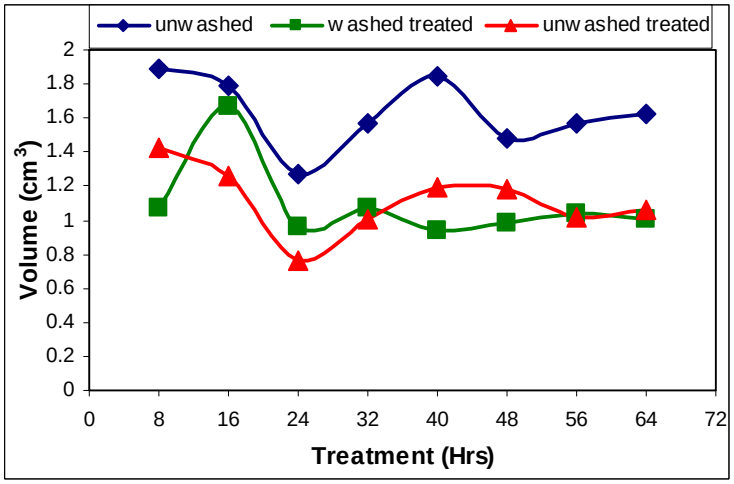


Fig 4.5 Volume change of 30 % unwashed, unwashed treated and washed treated fiber composites under steam environment.

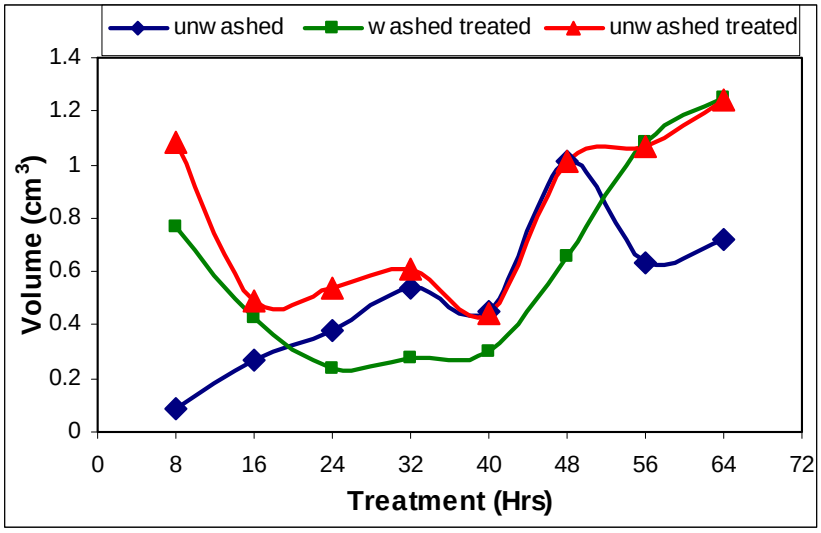


Fig 4.6 Volume change of 30 % unwashed, unwashed treated and washed treated fiber composites under saline environment.

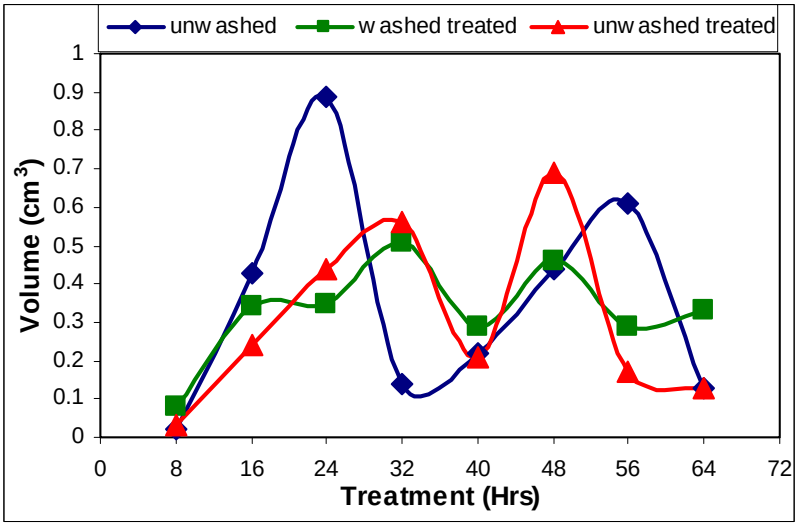


Fig 4.7 Volume change of 30 % unwashed, unwashed treated and washed treated fiber composites under subzero environment.

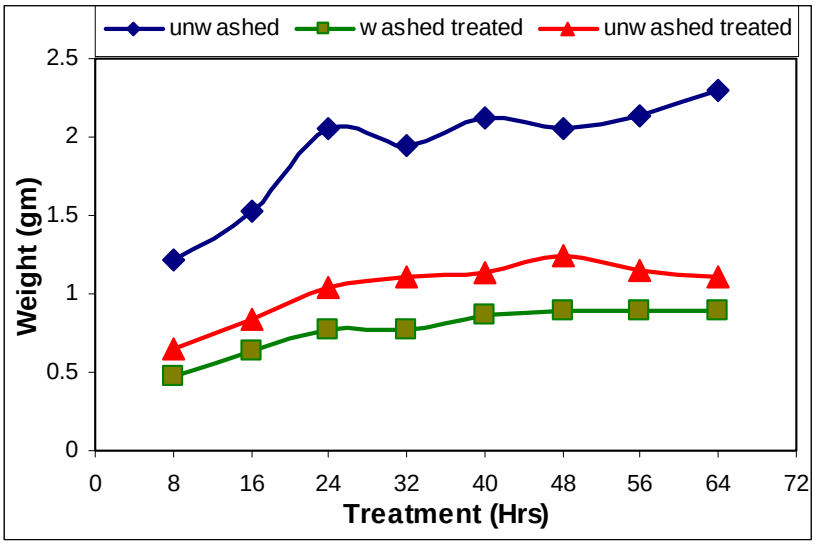


Fig 4.8 Weight change of 30 % unwashed, unwashed treated and washed treated fiber composites under steam environment.

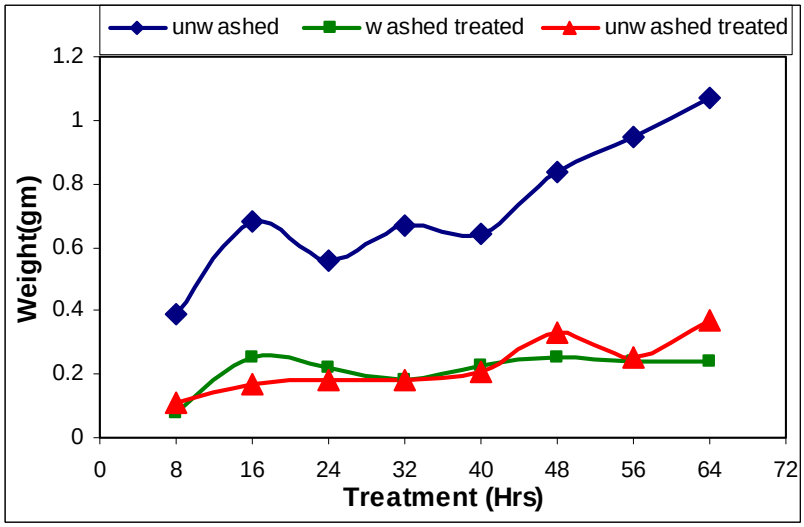


Fig 4.9 Weight change of 30 % unwashed, unwashed treated and washed treated fiber composites under saline environment.

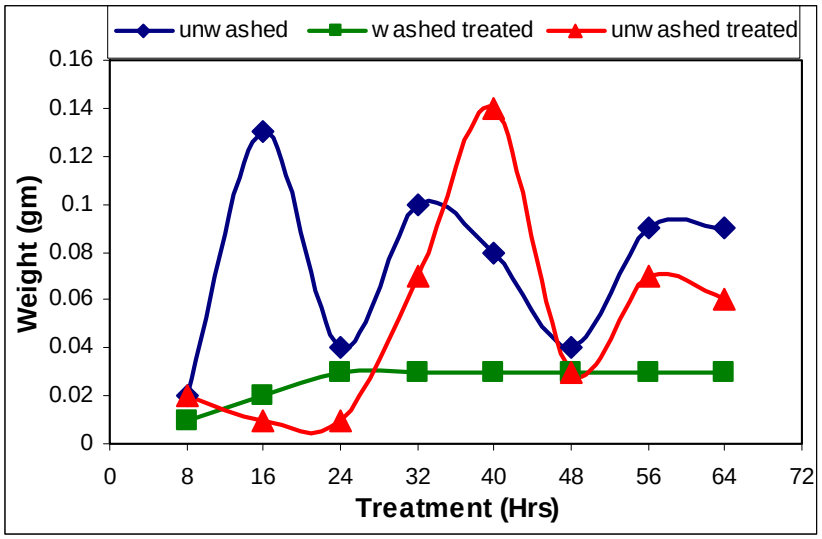


Fig 4.10 Weight change of 30 % unwashed, unwashed treated and washed treated fiber composites under subzero environment.

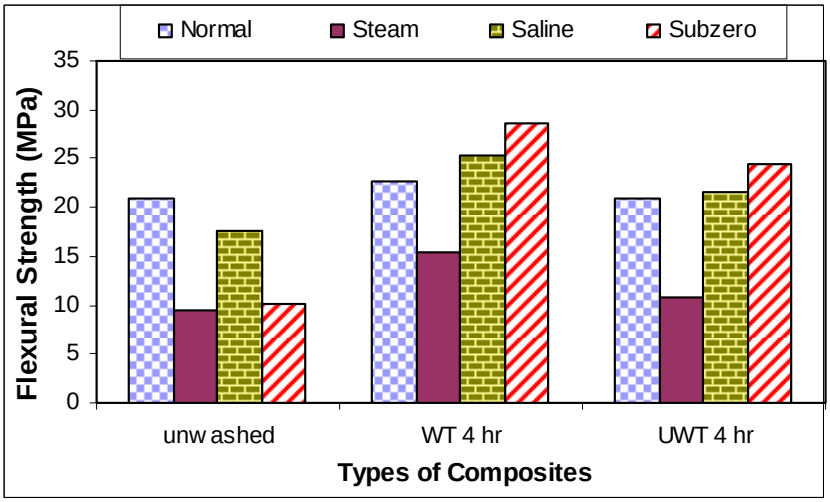


Fig. 4.11 Comparison of flexural strength of unwashed, unwashed treated and washed treated fiber composites under steam, saline and subzero conditions

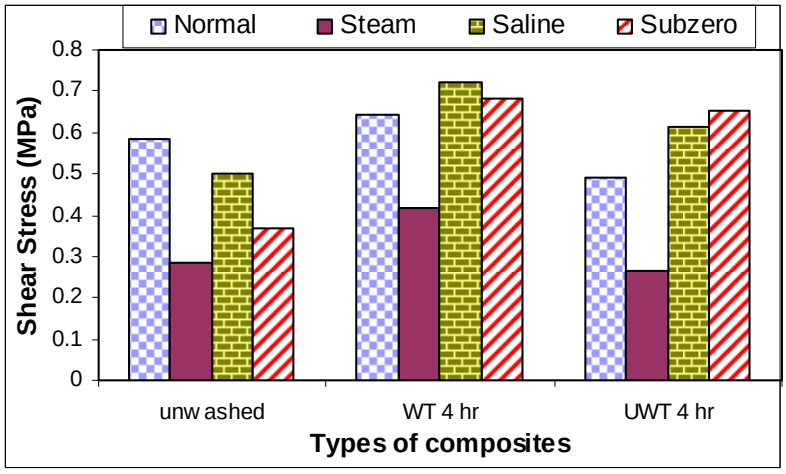
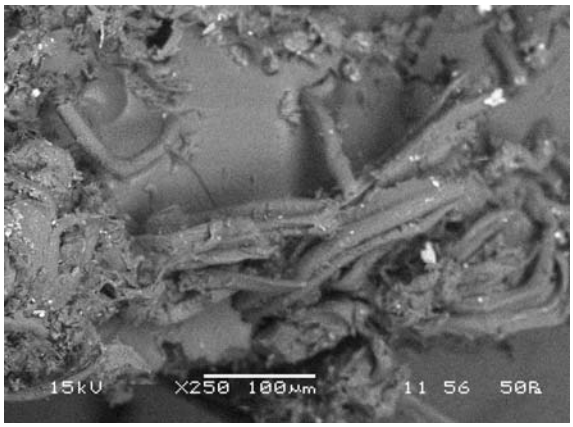


Fig. 4.12 Comparison of shear stress of unwashed, unwashed treated and washed treated fiber composites under steam, saline and subzero condition



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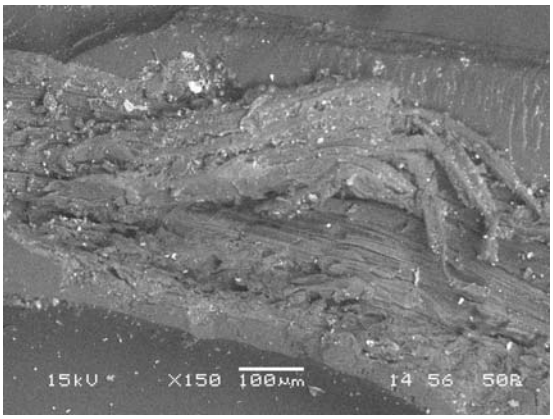
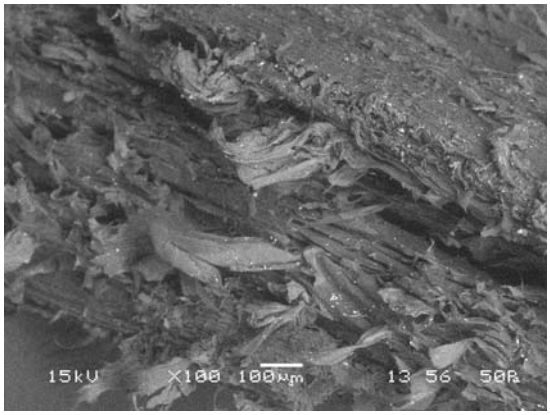


(b)



(c)

Fig 4.13 The fracture surface of the samples treated with alkali subjected to (a) Steam (b) Saline (c) Subzero treatment

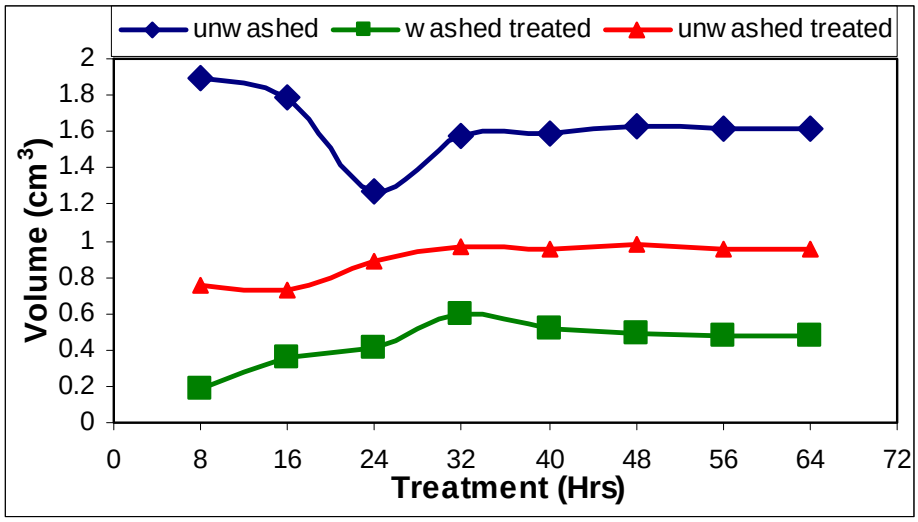


Fig.4.14 Cumulative volume change in different treated fiber composites for different time of exposure under steam treatment

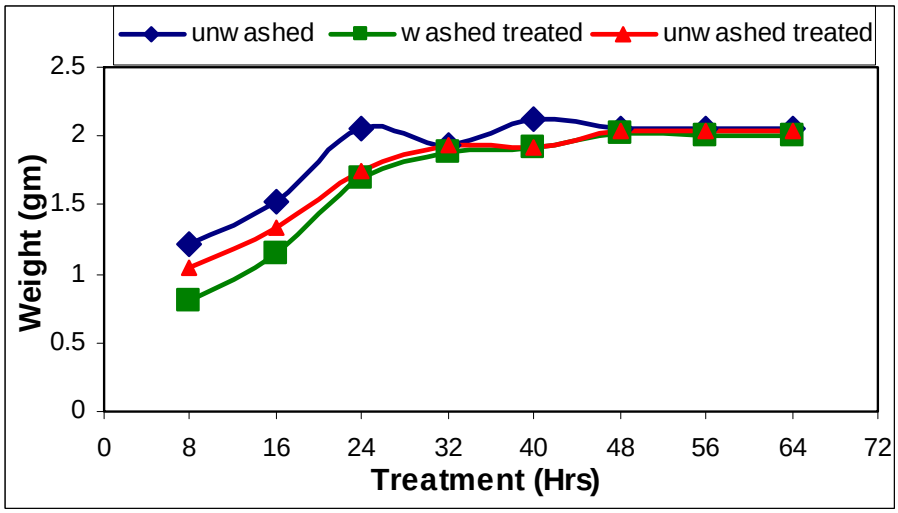


Fig.4.15 Time dependent cumulative weight change (due to % of moisture absorption) for different treated fiber composites exposed to steam

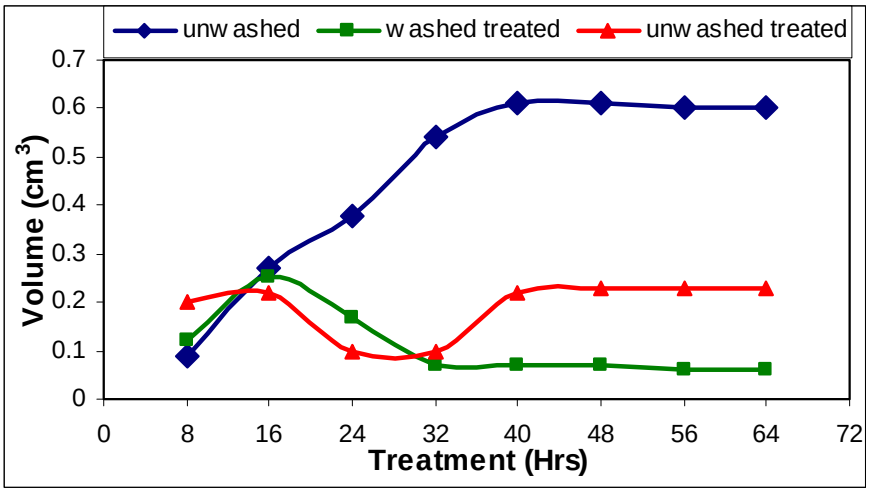


Fig.4.16 Cumulative volume change in different treated fiber composites for different time of exposure under saline treatment

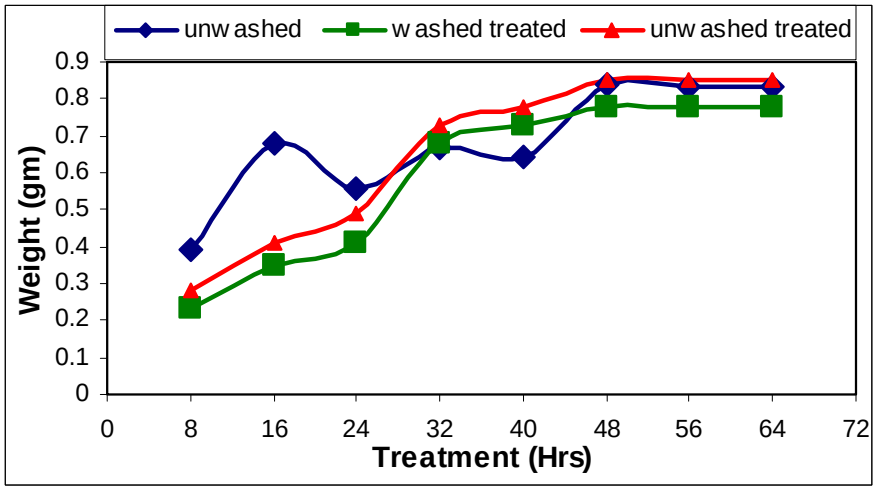


Fig.4.17 Time dependent cumulative weight change in different treated fiber composites exposed to saline treatment

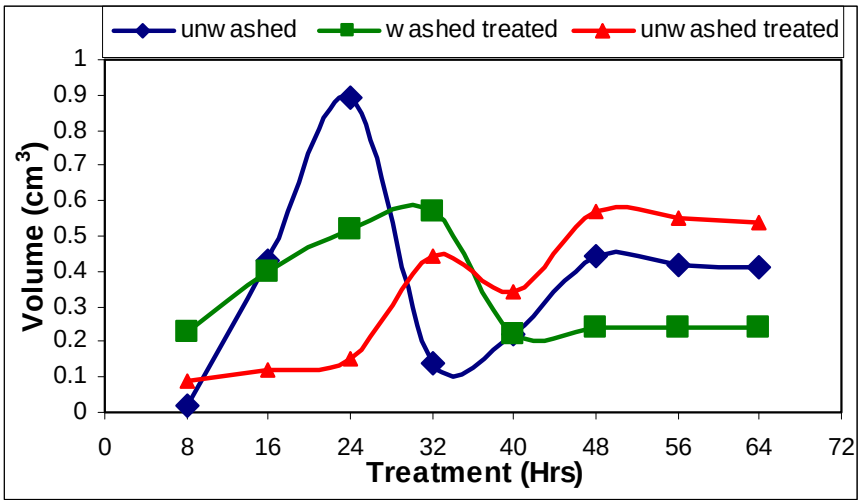


Fig.4.18 Cumulative volume change in different treated fiber composites for different time of exposure under subzero condition

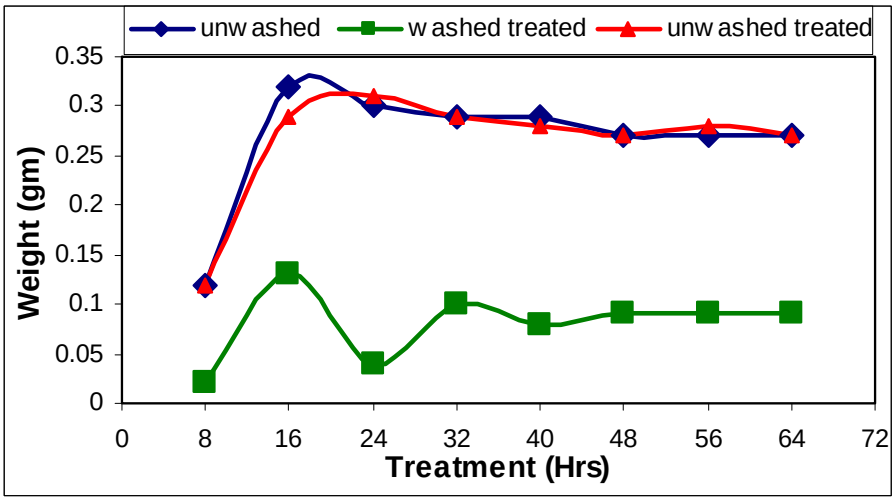


Fig.4.19 Time dependent cumulative weight change for different treated fiber composites exposed to subzero treatment

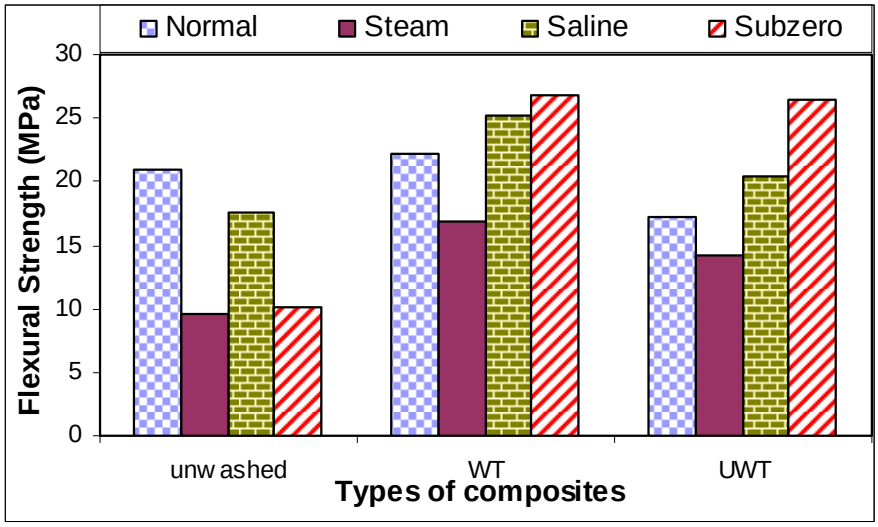


Fig.4.20 Variation of the flexural strength of the composites for various treatments

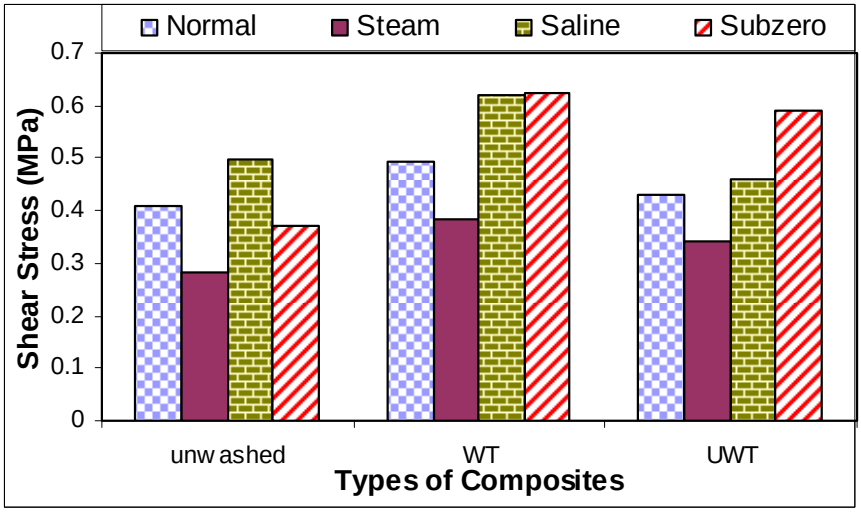
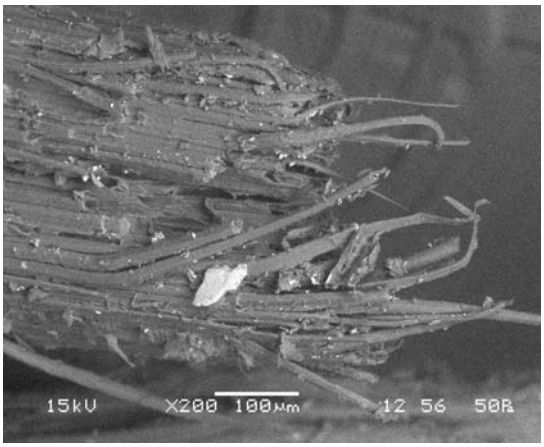
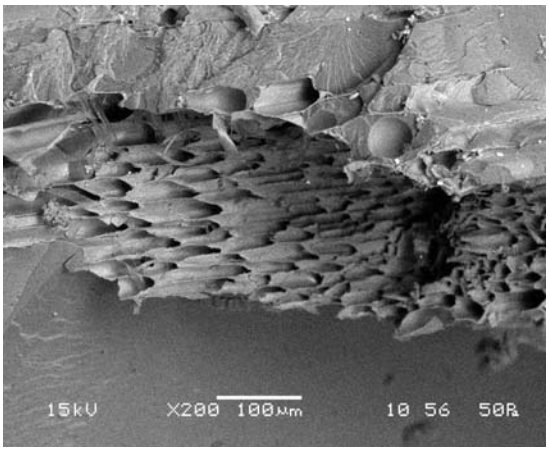


Fig.4.21 Variation of the shear stress of the composites for various treatments



(a)



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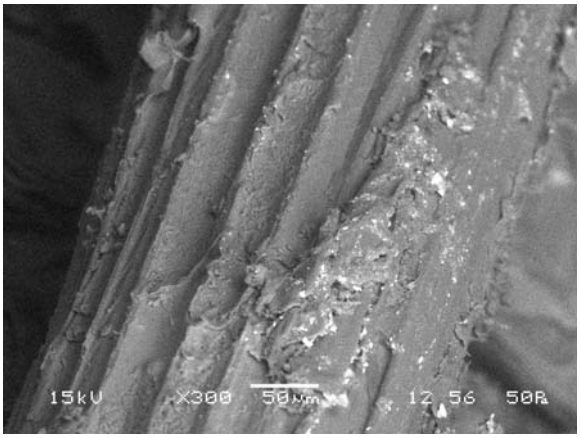

(c)

Fig 4.22 The fracture surface of the samples treated with acetone subjected to (a) Steam (b) Saline (c) Subzero treatment.

Chapter 5

CONCLUSION

CHAPTER-5

5.1 CONCLUSIONS

The following conclusions have been drawn from the above studies

1. The sugar cane residue bagasse an underutilized renewable agricultural material can successfully be utilized to produce composite by suitably bonding with resin for value added product.

2. By comparing the flexural strength of composite with varying fiber treatment, it was evident that best mechanical property results were obtained with bagasse fiber that were both washed and treated for both acetone and alkali treated fibers.

3. Water absorption capacity of the fiber found to be reduced when treated with alkali in comparison to acetone treated fibers. This may be due to the chemical reaction which reduces the hydroxyl group in the cell wall of natural fiber molecules, thus decreasing the water absorption capacity of the composite.

4. In general fiber pull out is the predominant mode of failure for all natural fiber composite. In our case from the morphology of the fractured surface (treated under different environment) for the alkali treated fiber it was found that fiber breakage were the predominant mode of failure. It is also found from washed and treated samples that after treating the fiber with alkali the fiber–matrix bonding has improved a lot which results in higher flexural strength.

5. For acetone treatment, fibers pullout were the predominant mode of failure. Therefore the strength in acetone treated fiber is some what low in comparison to alkali treated fiber. However it is established that fiber matrix bonding has improved a lot by chemical modification in comparison the untreated fibers.

6. From the preliminary study, the present work has shown promising results for these room temperature cured polymer matrix bagasse waste reinforced composites. The homogeneous characteristics of the fabricated composites as well as the level of their

mechanical properties enable them to have practical applications similar to those normally associated with wooden agglomerates.

5.2 RECOMMENDATION FOR FURTHER RESEARCH

From this work it is found that chemical modification of the fiber with alkali and acetone decreases the water absorption capacity of the fibers. Improvements in the process of bagasse cleaning and surface treatment could increase the performance of the composite and provide better competitiveness with respect to other materials in the same structural class. Chemical modification of the fiber surfaces such as Silane Treatment, Acetylation Treatment, Benzoylation Treatment, Acrylation Treatment Isocynates Treatment, Permanganate Treatment, Maleated coupling agents can be tried and a final conclusion can be drawn there after.

Natural fiber composites are showing promising results in tribological applications. The composite prepared can also be tried for tribological applications.

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Chapter 6

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